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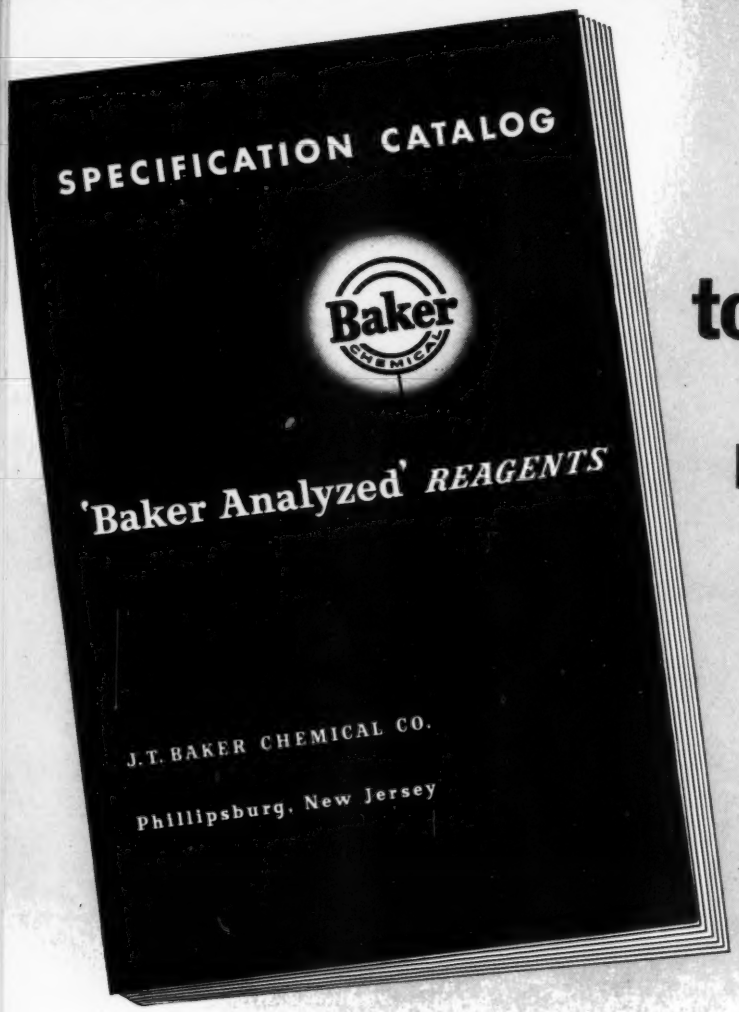

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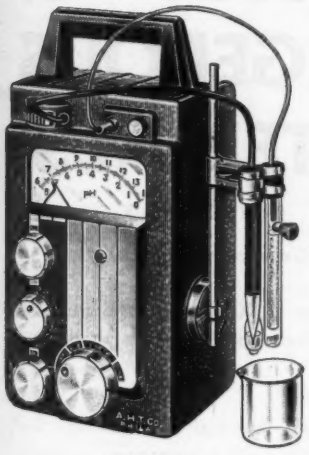

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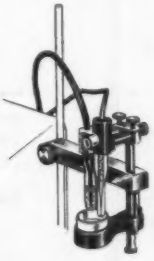

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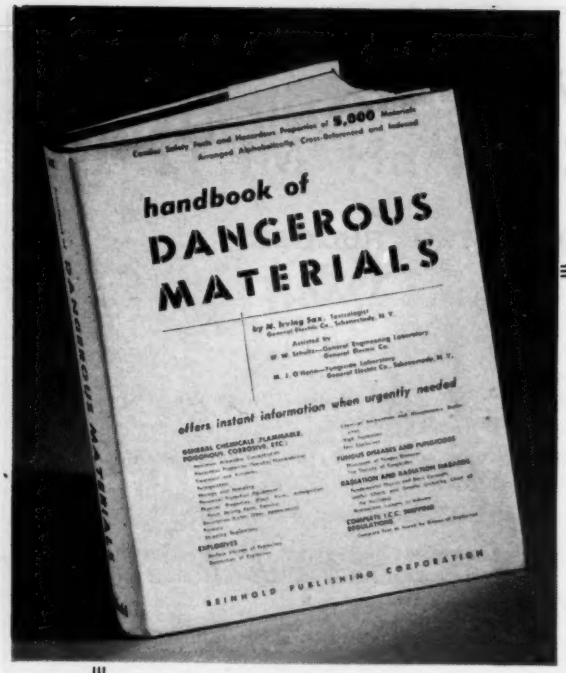

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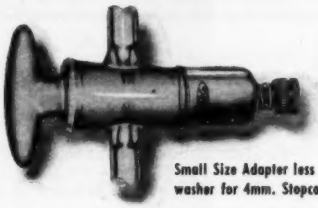

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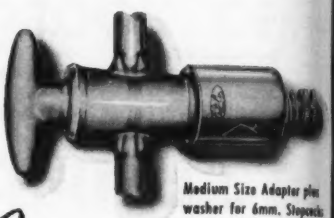

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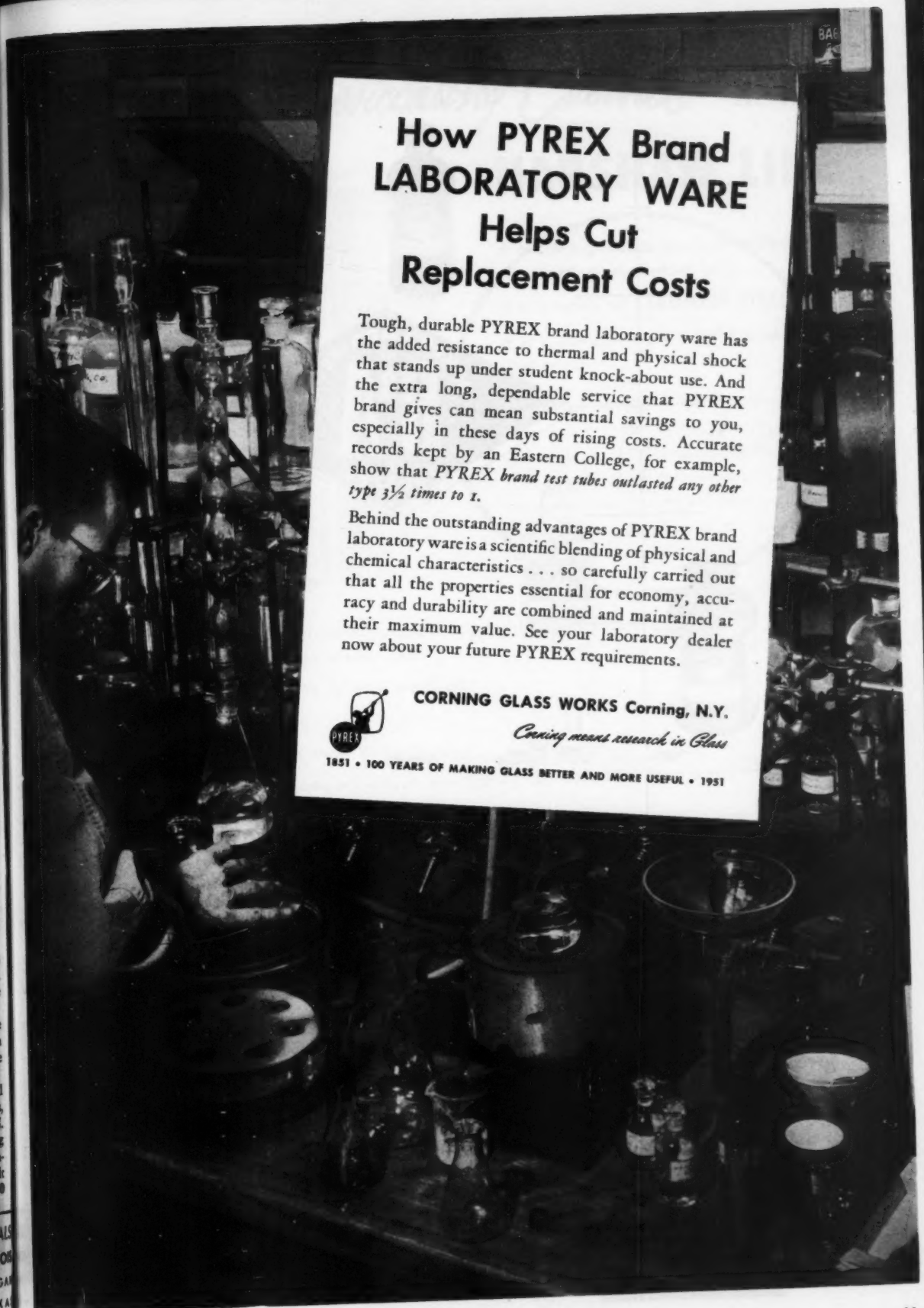
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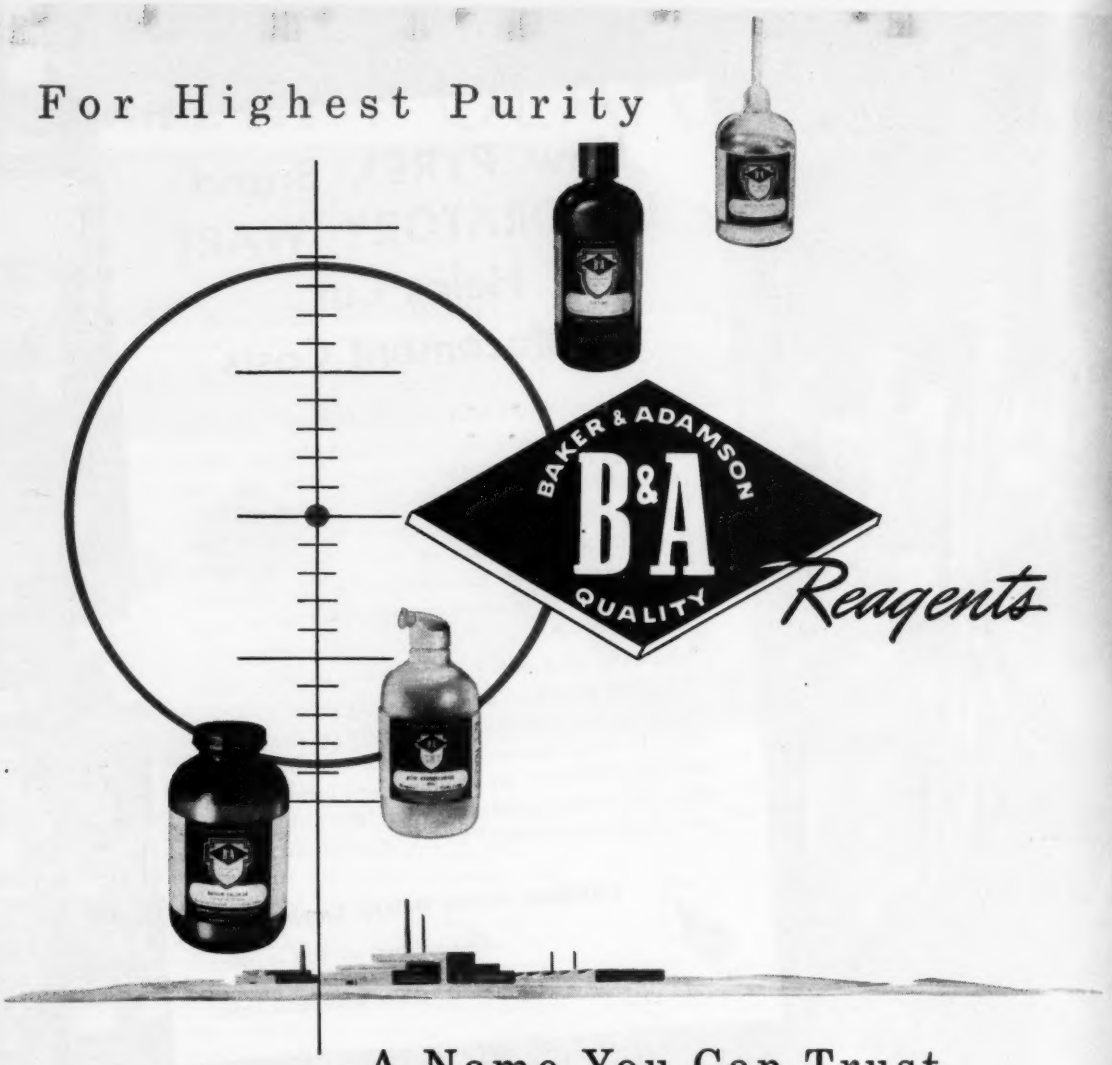

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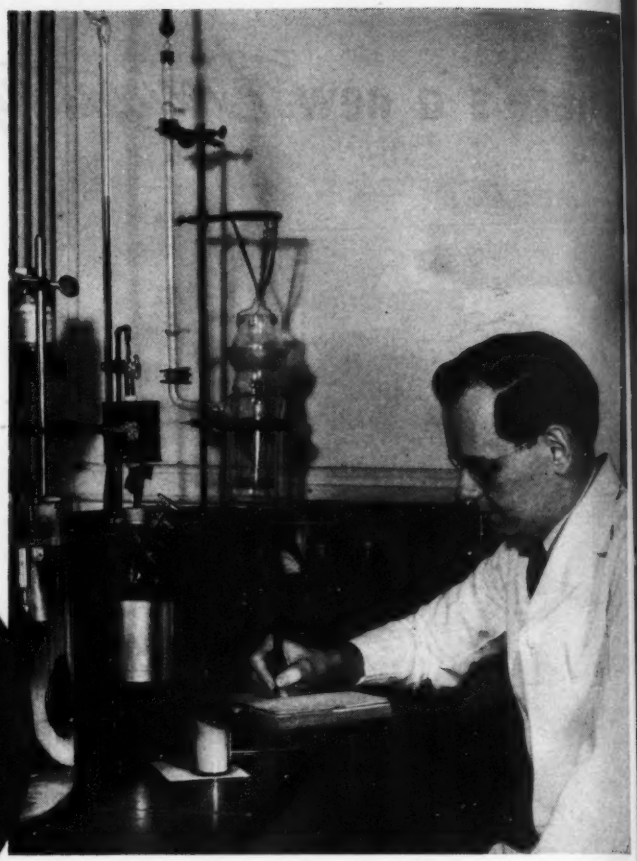

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1. Neuss, J. D., O'Brien, M. G., and Frediani, H. A., Analytical Chemistry **23,** *1332 (1951)*

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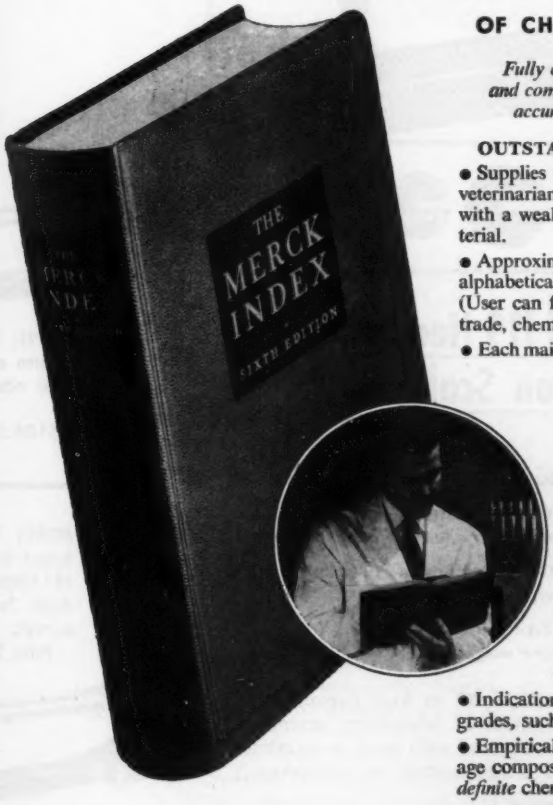

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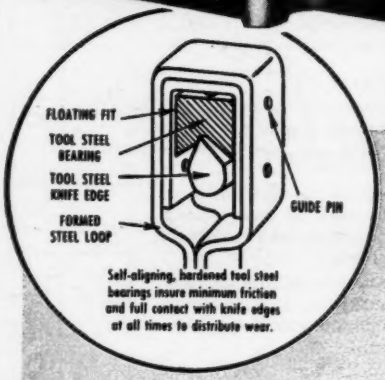

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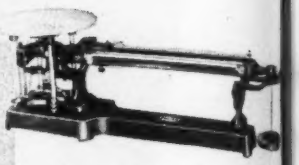

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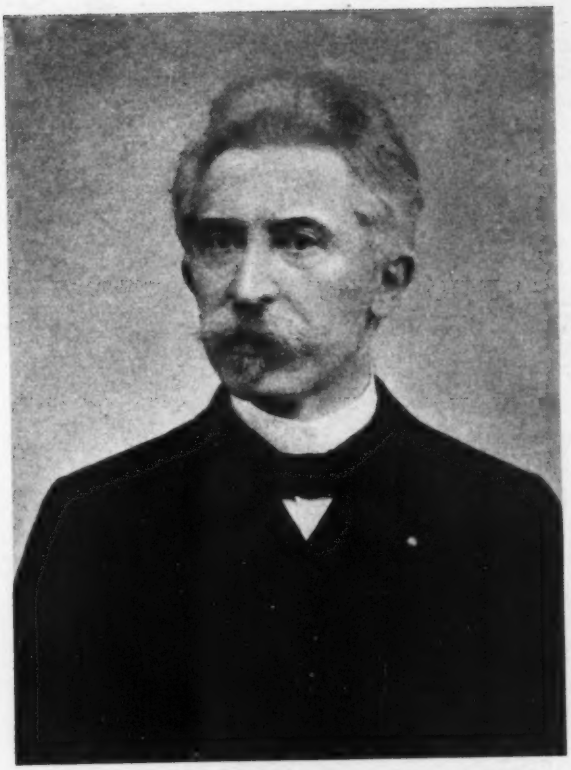

Johan Frederik Eykman

(See page 616)

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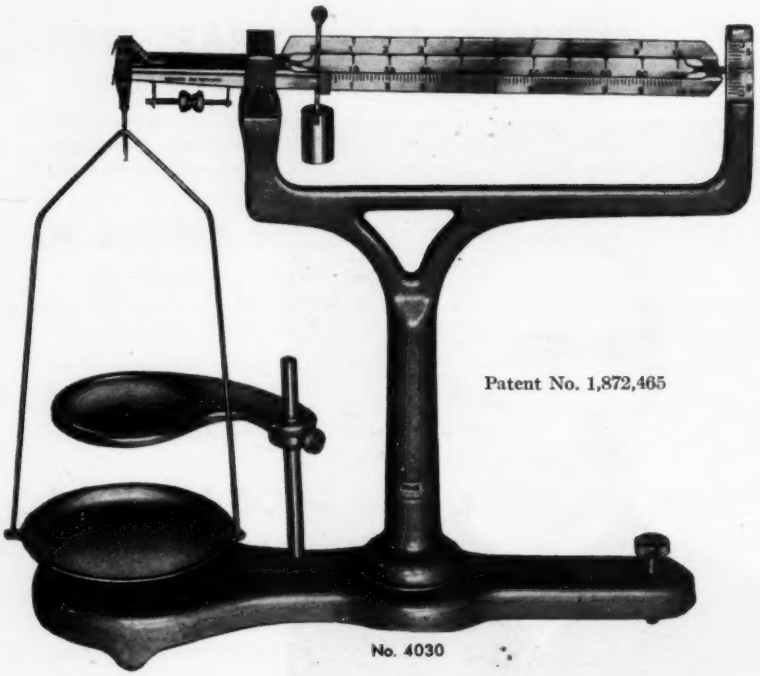

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Editor's Outlook

I HAVE just finished trying to tell my son one of the hardest things for children (and older folks) to understand: that our talents are not possessions for us to dispose of as we wish; that we have a responsibility to develop them, not merely in our own interest but for the benefit of everyone about us. If I have musical ability I need not become a professional musician but I should develop that ability so that everyone around me may be happier; if I have talent in art I should practice and make use of it so that I shall make the world around me brighter and more attractive; if I am a potential leader of others I have the responsibility to learn to do it unassumingly and effectively; if I have a keen and retentive mind it is my duty to sharpen it and put it to work, and not let it degenerate for lack of exercise. We have come to our present state of civilization and culture, not by everyone trying to see how nearly he can be like everyone else but by encouraging each person to make the most of himself and to exaggerate his differences to the utmost. If we want to have a fine garden we don't clip off the little budding shoots when they come through the flat ground; we encourage each one to grow as strong and as tall as it can, and after a while we have a beautiful garden or a rich crop of grain or a fine stand of timber.

It is hard for children—and the rest of us—to learn that we owe it to ourselves, to our friends and neighbors, and to our country to make the most of our talents and abilities. Still, the organization of our country—and a large portion of the rest of the world—is based upon this principle of social responsibility, while at the same time recognizing the rights of the individual in his own sphere.

The country is in an emergency in which it needs to use *all* its resources most effectively. From its name, it would seem that Selective Service had been founded upon the principle of social responsibility just outlined. To require each to serve his country according to his talents and abilities was its original intention. But for a long time it paid little if any attention to individual differences, except physical infirmities. More recently, it seemed that there was to be intelligent selection. Now it is reported that the head of Selective Service himself is suspicious of selection on any basis other than availability. This seems to many of us an attitude dangerous to the public interest.

The argument for conserving and developing the best brains has been presented so many times that it is unnecessary to repeat it. No one has been heard to deny that our country will survive—if it does—because of brains and ingenuity rather than population. Certainly, it will be hard to separate the brains out of the population, and very likely we cannot do it with perfect effectiveness and justice. But the first step in the accomplishment is obviously accepting the task in principle. And if we don't do *that*—we shall live to regret it.

JOHAN FREDERIK EYKMAN—A PHYSICAL CHEMIST[1]

H. S. VAN KLOOSTER
Rensselaer Polytechnic Institute, Troy, New York

JOHAN Frederik Eykman was born on January 19, 1851, in the small town of Nijkerk (Holland), the second son in a family with eight children—five boys and three girls. He and his brothers received their primary education from their father who kept a boarding school from 1850 to 1860. Although all five boys were unusually gifted, only one, Christiaan, rose to a position of pre-eminence as professor of hygiene at the University of Utrecht and winner of a Nobel prize in medicine (in 1929) for his work on beriberi. The family moved to Zaandam in 1860 where Johan Frederik, after completing his high-school education, was apprenticed to a pharmacist. On passing his preliminary examination in 1869, he spent two years in a pharmacy at The Hague and then went to Amsterdam to continue his pharmaceutical training at the Atheneum (since 1878, Municipal University) under Professor Gunning whose laboratory assistant he was for four years (1872–76). He obtained his diploma as pharmacist in October, 1874, and the next year he also passed the entrance examination to the University of Leiden where he planned to study physics and chemistry. He never went there, for in 1876 he was asked by the Japanese Government—no doubt on the recommendation of Gunning—to join a group of Dutch pharmacists who had been engaged to organize the hygienic and pharmaceutical services in Japan.

The shogunate had just been abolished and the modernization of Japan was in full swing, with the help and advice of numerous English, Dutch, German, and American experts. Eykman stayed nine years in Japan, the last four as professor of organic and pharmaceutical chemistry in the medical faculty of the University of Tokyo, at a monthly salary of 250 yen. He first taught in English with a Japanese interpreter but later on his lectures were given exclusively in German. With five hours of teaching a day, in addition to his phytochemical studies, his task was exceedingly onerous and he was forced to resign when his health began to give way. He developed stomach trouble which bothered him to the end of his life. Before he left he was received by the emperor who conferred on him the Order of the Rising Sun with the thanks of the Japanese Government for his services to the empire.

On his return voyage to his native land Eykman spent four months, from October, 1885, to February, 1886, at the world-famous botanical garden of Buitenzorg (Bogor), near Djakarta, the capital of Indonesia.

[1] Presented before the Division of History of Chemistry at the 119th Meeting of the American Chemical Society, Boston, April 2–6, 1951.

In Amsterdam, where Eykman settled, he finished his Commentary of the Japanese Pharmacopoeia, a voluminous work of over 1500 pages. Van't Hoff, at that time the leading physical chemist in Holland, extended a hearty welcome to Eykman, one year his senior, and gave him working space in his own private laboratory.

In 1890 Eykman was appointed professor of pharmacy at the University of Leiden. Since the laboratory and its equipment were in poor condition Eykman expected to be provided with modern facilities in a new location; the Dutch Government, evidently, did not accede to his legitimate requests, whereupon Eykman refused the appointment.

He then continued his researches in a private laboratory of his own until 1897, when the chair of pharmaceutical chemistry at the University of Groningen was offered to him on the death of the incumbent Plugge, who had been a collaborator of Eykman in Japan. This time he accepted and held this professorship until 1905. When his colleague Holleman left for Amsterdam Eykman took his place as professor of organic chemistry but remained in his pharmaceutical laboratory. He died ten years later of a heart attack, at the age of 64.

A perusal of the list of Eykman's publications, from 1878 to 1911 (about 60 in all), shows that Eykman was an extreme individualist. There is not a single paper showing double or plural authorship as a mark of close cooperation between master and pupils. Omitting Eykman's purely pharmaceutical and phytochemical researches carried out between 1878 and 1888, we find his first physicochemical investigations, performed in van't Hoff's laboratory, recorded in the early issues of the *Zeitschrift für physikalische Chemie*, founded in 1887 by van't Hoff and Ostwald. Eykman constructed a very simple and effective apparatus (depressimeter) for cryoscopic determinations with which he examined scores of organic compounds in their effect on the freezing point of various solvents. The anomalous results, obtained by Eykman in a number of solutions, led van't Hoff to the publication, in the same journal (*1*), of his celebrated article on solid solutions. About twelve years later Eykman designed and made an apparatus for ebullioscopic measurements, using the principle of heating the solution by means of the vapor of the solvent. This inexpensive outfit which can be made by anyone familiar with ordinary glass-blowing technique has been used for a number of years, both here and abroad.

An interesting study dealing with shikimi acid, a

polyhydric monobasic naphthenic acid derived from *illicium religiosum* (poisonous star anise, *shikimi-noki* in Japanese), deserves special mention because, as Ostwald (*2*) said in a review, "it is an excellent example of the applicability of physico-chemical methods for the determination of chemical constitution."

From 1890 onward Eykman devoted his time and energy almost exclusively to the study of organic compounds by means of refractometry. These researches have been edited in French under the title "Recherches réfractométriques" by his long-time friend and colleague Holleman. Upon this monumental work of 555 pages, published in 1919 and giving densities and refractive indices for close to 600 organic compounds, rests Eykman's lasting reputation.

At the very beginning of his refractometric studies Eykman found that the measurement of refraction (and dispersion) enabled him to distinguish between isomeric allyl and propenyl benzene derivatives. The allyl compounds when treated with boiling alcoholic potash changed invariably into the corresponding propenyl compounds with a considerable increase in molecular refraction (and dispersion). This important result, later supported by many corroborative experiments, led ultimately to the conclusion that it is useless to compute molecular refractions by means of so-called atomic refractions, a practice which is still continued.

Molecular refractions are obtained by multiplying the specific refraction by the molecular weight. The specific refraction, according to Gladstone and Dale's empirical formula, is $(n - 1)/d$; according to the formula derived by Lorentz in Leiden and independently by Lorenz in Copenhagen it is

$$\frac{n^2 - 1}{n^2 + 2} \times \frac{1}{d}$$

This specific refraction is stated to be independent of the temperature and to hold as well for the gaseous as for the liquid state. This is only approximately true; the first formula gives at higher temperatures values which are lower, and the second, values that are higher than those obtained at room temperature.

Eykman set himself the task of extending the range of operating temperatures. Perkin, Sr., (*3*) had already determined values for n and d up to temperatures of around 90°C. Eykman went up to 140°, thus covering an interval of 120°. Without impairing the accuracy of his measurements he was able to reduce the size of his hollow glass prism containing the liquid to such an extent that about 2 ml. were sufficient to determine density and refractive index. The prism, closed on either side by a thin cover glass, contained a tiny thermometer of his own construction. Eykman's special thermometers, each covering only a few degrees in order to do away with the stem correction, were so skilfully made that they could not be duplicated by professional glass blowers of a noted supply company in Germany. The prism in Eykman's refractometer was movable while collimator and telescope were kept in a fixed position at an angle of 40°. This arrangement has the advantage that only small openings in the heating bath are necessary to allow the passage of the light from the source. There is then no minimum deviation but two positions of the prism can be found to which the incident ray is deflected in such a way that it passes through the cross section in the telescope. Using this instrument Eykman found that between 20 and 140°C. the Gladstone-Dale expression deviated between 0.6 and 0.8 per cent and the Lorentz-Lorenz formula between 0.7 and 1.3 per cent. He succeeded in setting up an empirical expression

$$\frac{n^2 - 1}{n + 0.4} \times \frac{M}{d}$$

for the molecular refraction which gave for a large number of compounds a deviation not exceeding ±0.1 per cent, a remarkable improvement over the expressions of his predecessors.

Following the example of Nernst, who first deprecated the use of Eykman's formula since it lacks theoretical justification (*4*), practically all physicists and chemists have used the Lorentz-Lorenz formula. Holleman was, for a long time, the only chemist who recommended Eykman's equation. "Why," asks Holleman, "reject a good dinner because the cook has not given us his recipe?"(*5*) After all, there are numerous empirical expressions in physical chemistry, such as Dühring's rule and Trouton's rule, which are used constantly. Even the theory of the parachor as developed by Sugden is based on an empirical relation found by McLeod. Eykman himself, unable to convince his contemporaries of the superiority of his equation, used the older expressions along with his own.

The net result of Eykman's researches is a complete repudiation of the scheme developed by Brühl, Eisenlohr, and their followers, of computing molecular refractions with the aid of a set of fixed values for atomic refractions, double and triple bonds, conjugated double bonds, etc. This is rather easy but does not always lead to reliable results. Eykman's method, based on a comparative study of analogous compounds, saturated and unsaturated, is undoubtedly more complicated but at the same time more convincing.

In 1915 the writer, from 1906 to 1910 a student under Eykman, expressed the hope that future generations might appreciate Eykman's work more than his contemporaries (*6*). In going through the literature dealing with refractometry over the past 35 years, it was, therefore, gratifying to find that Eykman's formula has been rescued from oblivion and its usefulness extended by several American scientists whose work is briefly reviewed below.

In 1936 and 1937 Kurtz and Ward (*7*) showed that the theoretical foundation of the Lorentz-Lorenz formula is unsound. They found that the Sellmayer-Drude dispersion equation can be modified so that it will give data which are in quantitative agreement with Eykman's empirical equation. Furthermore, these authors pointed out on the basis of previous work that the Eykman equation also applies to the effect of pressure on

the relation between density and refractive index. This was strikingly confirmed the next year by Gibson and Kinkaid (*8*) who subjected benzene at temperatures between 25° and 65°C. to pressures up to 1200 bars. Using the Maxwell relation

$$n^2 = \epsilon$$

Kurtz and Ward replaced the Clausius-Mosotti expression by the modified Eykman equation

$$\frac{\epsilon - 1}{\sqrt{\epsilon} + 0.4} \times \frac{M}{d}$$

and here, also, it was found that the latter represents the experimental data much better than the Clausius-Mosotti expression. For example, for a nonpolar hydrocarbon, 2,2,4-trimethylpentane, Smyth and Stoops (*9*) found the molar polarization to be 39.14 at −110°C. and 39.86 at 90°. Using Smyth and Stoops' data with the modified Eykman equation the writer obtained the values 86.91 and 87.15, a difference of only 0.3 per cent over an interval of 200°.

Quite recently Dreisbach (*10*) showed that over the short range of 20° to 25°C. the Eykman equation represents experimental data for a wide variety of organic compounds more accurately than the Lorentz-Lorenz formula does. Last year Kurtz, Amon, and Sankin (*11*) supplied tables for hydrocarbons and nonhydrocarbons which clearly indicate the accuracy of the Eykman equation over a wide range of temperatures.

For one who has seen and heard both Lorentz and Eykman, it is clear, apart from all other considerations, why Eykman's formula was rejected by his contemporaries and Lorentz' formula universally accepted. Lorentz was renowned as a scientist, as a teacher, as a man among men. He was a brilliant lecturer, his students adored him, his friends were legion. Eykman, on the other hand, though gifted as a scientist and unexcelled as a glassware manipulator (van't Hoff used to say that Eykman had learned this technique from the famous Japanese sorcerers), made himself obnoxious by his biting sarcasm and his perpetual contrariness. Van't Hoff, highly appreciative of Eykman's talents, found it difficult to satisfy the demands to which he was subjected, in view of the limited budget under which he operated his laboratory. When a student examining a substance given to him by van't Hoff went to Eykman for advice, he was told to get Kahlbaum's catalogue of reagents and look up the cheapest substance offered for sale. That, no doubt, would be the unknown. Eykman disliked lecturing. One hour a week was all he ever put in on this part of his official duties. He used to tell his students to study the various topics for themselves and report to him when they had done so. Being always contrariminded, he made himself impossible among his colleagues and for that reason, if for no other, he was never elected a member of the Dutch Royal Academy of Sciences. His one outstanding hobby—for which he was known throughout the city of Groningen—was his love of horses. Whenever the weather was favorable he could be seen riding in his dogcart or walking on the sidewalk with his horse pulling the cart and keeping pace with his master. Of commanding height, always immaculately dressed in a double-breasted frock coat, he had the respect, if not the affection, of those who knew him. Under an austere exterior he hid a fondness for the children of the indigent in the immediate vicinity of his laboratory which was located on the outskirts of the city. On the eve of Saint Nicholas (December 5) his factotum, the laboratory custodian Roosjen, was always instructed to distribute the gifts from the good Saint, with strict orders not to divulge the name of the donor. Of his private life little was known, except that he had married the sweetheart of his youth who for nine years had patiently awaited his return from Japan. The marriage, however, proved to be a failure and Eykman, for the rest of his life, lived alone having few outside contacts and very few students. Toward the end of his life he once remarked in a bitter moment that his horse was the only friend left to him. He was his own worst enemy and as a result his life was not a happy one.

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THE Board of Editors of "Inorganic Syntheses" has announced that the copy for Volume IV of that publication will go to the publishers during the early part of 1952. However, syntheses are still being accepted and will continue to be accepted for some time yet. Any which are accepted too late for Volume IV will be held over for Volume V. The Editors are particularly anxious to publish methods of preparing important or particularly interesting inorganic compounds, including the preparation of compounds of the less familiar elements, radioactive elements and some of the elements which have attracted attention in recent years such as fluorine, germanium, and beryllium.

Methods which have been published in chemical journals may still be published in "Inorganic Syntheses" if they can be described to advantage in greater detail than in the journal or if new and pertinent information can be added.

It should be pointed out that while "Inorganic Syntheses" is being used by some courses in inorganic preparations, it is intended primarily as a manual for research workers.

Until further notice, manuscripts should be sent to Dr. John C. Bailar, Jr., Department of Chemistry, University of Illinois, Urbana, Illinois. Directions to authors and style sheets will be sent upon request.

STRUCTURAL CHEMISTRY OF THE INTERHALOGEN COMPOUNDS

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THE discovery and preparation of several of the interhalogen compounds followed shortly after the discovery of the elements themselves. Since the halogens are all relatively strongly electronegative elements, each lacking one electron to complete its outer shell, they form diatomic molecules with a shared electron-pair bond between them:

In a very similar manner, interhalogen molecules are formed, the simplest type being represented by ClF, BrCl, IBr, etc., whose physical properties are intermediate between those of the two elements involved (*1*) (see the table and Figures 1 and 2).

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	F_2	ClF	Cl_2	$BrCl$	BrF	Br_2	ICl	IBr	I_2
M. p., °C.	−223	−156	−102	−66	−33	−7	27	36	114
B. p., °C.	−187	−100	−35	5	20	59	97	116	184
Sp. gr.	1.31	1.62	2.49	..	..	3.12	3.18	4.4	4.9

However, these properties are not necessarily the average of those of the two parent elements.

Of the six possible uni-univalent halogen halides, five, all except iodine fluoride, are known to exist; the latter is probably too unstable, since in the known iodine-fluorine compounds, iodine always has a valence greater than 1 (*2*).

Of considerably more interest from a structural standpoint are the interhalogen compounds in which one of the halogens has a valence greater than 1. Three such series exist: AB_3, AB_5, and AB_7. No compounds are known where an even number of atoms of one halogen combine with an odd number of another; such a molecule would have an unpaired electron.

Of the twelve possible tervalent halogen halides only four are known to exist. Prideaux (in 1906) predicted the formation of IF_3, as well as IF, but since he claimed that BrF could not be formed because he always obtained BrF_3 from various proportions of the two elements, his predictions are

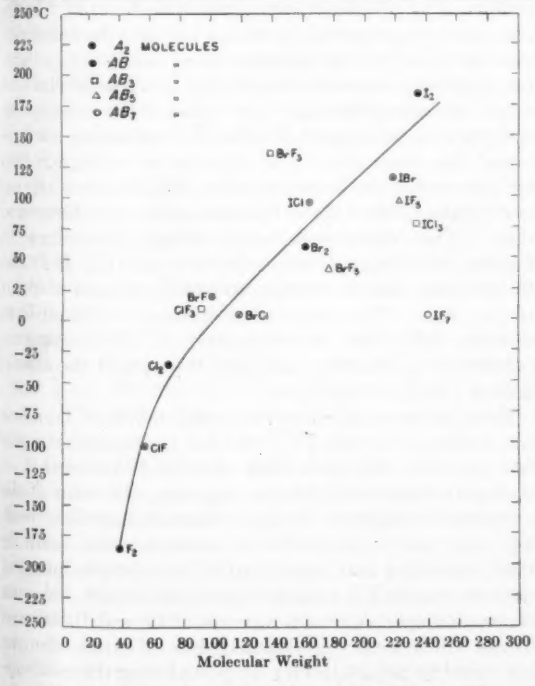

Figure 1. Boiling Points of the Interhalogen Compounds

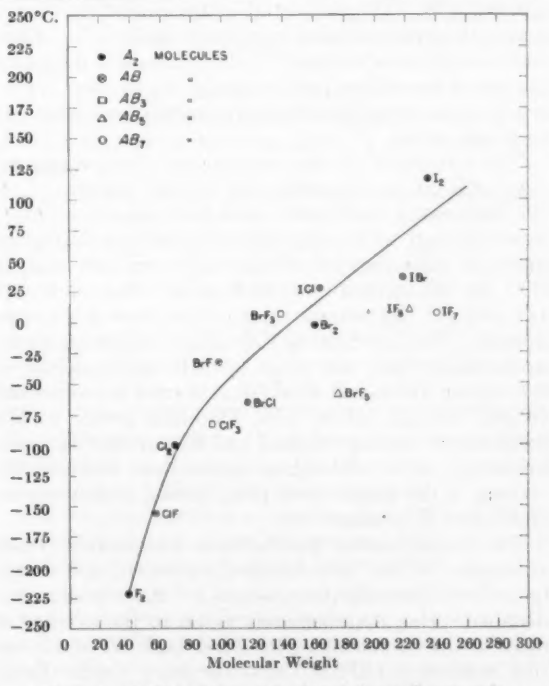

Figure 2. Melting Points of the Interhalogen Compounds

probably not reliable (*3*). More recent measurements of the vapor pressures and melting points of mixtures of iodine and iodine pentafluoride show no evidence of the formation of any iodine fluorides in which iodine has a valence of less than five; direct combination of the elements also produces only the penta- and heptavalent compounds (*4*).

Similarly, of the twelve possible quinquevalent compounds, only two, BrF_5 and IF_5, are known and of the septavalent, only IF_7 (*2, 4, 5, 6*). IBr_5 is listed in "The Condensed Chemical Dictionary" (*7*), where it is described as a brown liquid, but since no further reference has been found to such a compound, it may be merely a mixture. No compound of the halogens containing three or more different elements is known.

The interhalogen compounds are all prepared either from direct combination of the elements or by halogenation of a lower halogen halide, usually at temperatures from about 200° to 250°C. and often with special precautions and equipment because of the extreme reactivity and corrosiveness of many of these compounds (*3, 4, 8, 9*). The polyfluorides are becoming increasingly useful as substitutes for fluorine in fluorination reactions, chlorine trifluoride being even more reactive than fluorine itself, probably due to its dissociation to form fluorine atoms. Iodine heptafluoride is also more reactive than either fluorine itself or the pentafluoride, possibly due to the catalytic effect of the I atom (*10*), but it is less hazardous to handle and transport. Iodine monochloride has long been known as an analytical reagent and catalyst, and is now also used in organic syntheses; it is also used medicinally, as is the trichloride (*7*). The compounds of bromine with chlorine and with iodine are also used medicinally (*7*). Chlorine monofluoride was used by the Germans in the manufacture of incendiary bombs during the last war. Several of the compounds also have potential military uses as poison gases.

The structures of the interhalogen compounds are very difficult to determine for several reasons: (1) the lightness of the fluorine atom prevents any accurate measurements of its compounds by electron diffraction methods, since fluorine diffracts electrons only slightly (*11*), (2) the instability of some of the compounds (*10, 11*), and (3) the extreme reactivity of most of the compounds. The mechanical difficulties of making measurements are thus very great (*9*). It was found necessary to use Teflon cell windows at normal pressures and to seal the salt prisms with Fluorlube grease at low pressures in making infrared and Raman spectra measurements; even with these precautions considerable etching of the prisms took place during measurements of IF_5 and IF_7 spectra (*10*).

The uni-univalent interhalogen compounds, being analogous to the homohalogen molecules, are simple linear molecules with the structure $A—B$, the interatomic distances being approximately equal to the average of those of the two elements involved (*12*). The absorption spectra of ClF, ICl, and IBr have similar forms (*13*).

Chlorine monofluoride is a nearly colorless gas, which condenses to a yellowish liquid and solidifies as a white solid. It was first prepared by Ruff and Ascher in 1928, although the possibility of its existence was predicted by Prideaux in 1906 (*3*). It dissociates to form chlorine and fluorine atoms, the dissociation energy being either 58.9 or 61.2 kg.-cal./mol, depending upon whether the F or the Cl atom produced by the dissociation is in an excited state (*9*). It dissolves in liquid chlorine without further reaction.

The bond energies of the interhalogen compounds compared with the bond energies of the component halogens are a measure of the extent of ionic character of the molecule. Thus Pauling (*14*) has calculated the bond energies of the compounds from the formula $^1/_2$ ($A—A$ + $^1/_2$ ($B—B$), where $A—A$ and $B—B$ are the bond energies of the individual halogens. On comparing these calculated bond energies with the measured bond energies, he found ClF to be about 30 per cent ionic, *i. e.*, the ionic character of ClF is greater than that of HCl. The internuclear distances of ClF were found by electron diffraction to be 1.65 ± 0.01 Å., agreeing closely with the results obtained by spectrographic measurements (*15*). The calculated distance from the sum of the atomic radii of each element is 1.67 Å. (*16*). In common with the other interhalogen compounds, relatively little is known about the mechanism of the reactions of ClF, due to its extreme reactivity and the difficulty of isolating the final products (*9*).

Bromine monofluoride is a pale red gas, less stable than chlorine monofluoride. It was also first prepared by Ruff, in 1933 (*9*), although its existence had been predicted by Prideaux many years earlier (*3*). It decomposes spontaneously at about 50° into the trifluoride and free bromine, the reaction being catalyzed by light. Its chemical properties are similar to those of the bromine polyfluorides (*4*). Its ultraviolet absorption spectrum shows a band at 3250 Å., indicating a vibrational fine structure. The dissociation energy is 50.3 kg.-cal./mol if the bromine atom released is in the excited state, or 59.9 if the fluorine atom is in the excited state. The calculated bond energy according to Pauling (*14*) is approximately 60.6, so that BrF also shows some ionic character, up to a maximum of about 16 per cent. The internuclear distance as found from electron diffraction measurements is 1.78 Å. as compared to a calculated value from the sum of the atomic radii of 1.81 Å. (*15, 16*).

Bromine monochloride is a reddish-yellow liquid or gas, boiling at about 5°C. A clue to its existence was first noted by Balard in 1826 when he observed that on mixing bromine and chlorine together, the color of the bromine diminishes. A phase diagram based on freezing point and vapor pressure measurements, made in 1907, showed a continuous curve, but definite proof of the existence of BrCl was not given until 1928, when the absorption spectrum of a mixture of Cl and Br showed bands which were not characteristic of either element but could be accounted for by postulating the existence of BrCl. In the solid form it forms a series of solid

solutions with both chlorine and bromine, a reflection no doubt of the close similarity between the two kinds of atoms (*2*, *4*). BrCl is typically covalent, having only about 1 per cent ionic character (*14*).

Iodine monochloride was discovered by Davy and Gay-Lussac in 1814. It is interesting to note that Liebig a few years later missed the opportunity to become the discoverer of bromine by confusing it with this compound. He had found iodine in the residues from the manufacture of salt from sea water and had observed that the resulting starch iodide turned bright yellow on standing overnight. He saturated some of the saline mother liquor with chlorine and distilled off a liquid which also turned starch yellow. Knowing of the existence of iodine monochloride, which reacted similarly with starch, he assumed that he had also prepared this compound and promptly forgot about it—until he received Balard's paper several months later describing almost identical experiments by which Balard had isolated not iodine monochloride, but a new element, bromine.

Iodine monochloride occurs in two forms, red cubic needles which melt at 27° to a dark red oily liquid (α form), and red-brown, rhombic, six-sided plates melting at 14° (β form). Both crystalline states appear almost black unless observed in very thin layers. When chlorine is passed over iodine a dark red liquid forms which solidifies on standing to the unstable β form. On further standing it is converted to the stable α form, which remains stable under all conditions. Below 14° either α or β crystals may separate out, depending upon whether an α or β crystal is added. On cooling to −10°, the β form is obtained. Since the β form changes spontaneously to the α form at room temperature, only the α form exists at the boiling point (97°), regardless of the original form (*8*). Iodine monochloride is the only interhalogen compound showing polymorphism, but so far the structure of the solid compound has not been studied (*9*).

On heating ICl it decomposes to the elements. The calculated and observed bond lengths are in agreement. The absorption spectrum is similar to that of ClF. The bond in ICl appears to be about 8 per cent ionic in character (*14*). The molecular dipole moment is 0.65×10^{-18} e. s. u. (*17*).

Iodine monobromide occurs as dark gray crystals, similar to iodine. It was first prepared by Balard in 1826 (*9*). The calculated and observed bond lengths agree (*17*). The bond appears to be about 4 per cent ionic in character.

It has also been found that when IBr is incorporated in a film of an organic polymer such as starch, its absorption coefficient increases, a fairly strong band appearing in the visible spectrum. IBr under these conditions shows dichroism, and X-ray diffraction patterns show a new pair of diffuse lines, indicating diffraction by independent line gratings, such as might be formed by straight chains of halogen atoms. The periodicities of the halogen chains are independent of the periodicity of the original polymer (2.90 Å. for IBr, less than the 3.90 Å. distance between the unbonded atoms, but more than the 2.70 Å. distance of the single intramolecular bond). These shortened distances between known atom pairs may mean either hydrogen bonding, bonding of both atoms to a central atom, or a direct bond between the two atoms, this latter probably being the case with the IBr molecule. Bonds are formed at 180° to each other and are weaker than single covalent bonds but much stronger than van der Waals forces. Such compounds are therefore called linear polymeric halogens (*18*).

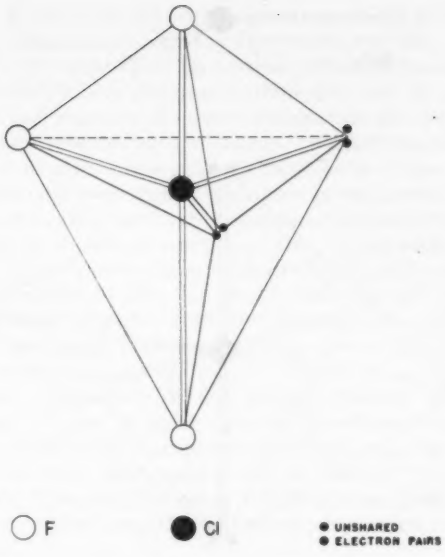

Figure 3. Structure of Chlorine Trifluoride

The polyvalent halogen compounds differ from the univalent compounds in that the physical properties do not necessarily lie between those of the parent compounds:

	F_2	ClF_3	Cl_2	BrF_3	Br_2	ICl_3	IBr_3	I_2
M. p., °C.	−223	−83	−102	5	−7	33	...	114
B. p., °C.	−187	11	−35	135	59	77	...	185
Sp. gr.	1.31	1.77	2.49	2.8	3.12	3.12	...	4.93

	F_2	BrF_5	Br_2	IF_5	IF_7	I_2
M. p., °C.	−223	−61	−7	8	5–6	114
B. p., °C.	−187	41	59	97	4 sub.	185
Sp. gr.	1.31	2.47	3.12	3.5	2.8	4.93

This difference reflects the differences in their structures, which have been investigated in recent years by means of both infrared and Raman spectra.

Chlorine trifluoride is a colorless gas which condenses to a pale green liquid and freezes to a white solid. It was first prepared by Ruff and Krug in 1930 (*9*). It is very reactive, most metals and non-metals either reacting explosively or burning in the gas. It is typically covalent, as indicated by its low boiling point and practically negligible specific conductivity, less than 10^{-6} ohm^{-1} cm.$^{-1}$ (*19*). Its structure is still somewhat in

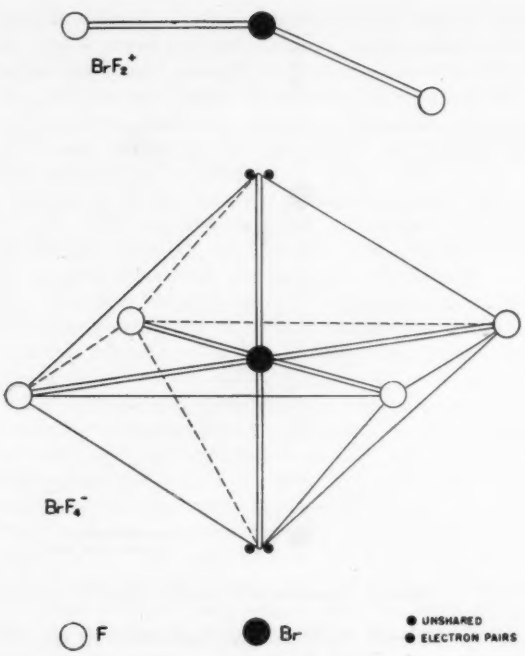

Figure 4. Structure of Bromine Trifluoride

doubt. Molecules in which an unshared pair of electrons occurs generally have a polyhedral structure in which the unshared pair occupies one of the corners of the polyhedron, replacing the shared pair of electrons of a bond (*14*). In an ion such as ICl_4^- the four chlorine atoms are located at the corners of a square with the iodine atom in the center. Two unshared pairs of electrons, one above and one below the ICl_4^- plane, give the complete structure an octahedral form; three unshared pairs in structures such as $ClICl^-$ are arranged in a plane about the I atom and perpendicular to the linear $ClICl^-$ structure, so that a trigonal bipyramid is formed (*14*). Chlorine trifluoride, having three shared electron pair bonds and two unshared pairs of electrons, may be considered as having a similar type of structure (Figure 3). If it is a planar molecule, the three shared bonds would be at the base corners of a trigonal pyramid about the Cl atom at the center and the unshared pairs of electrons would be at the apexes; if it is an asymmetrical pyramid, one of the shared bonds would be at the apex and the unshared pairs of electrons would be at two of the base corners. Electron diffraction measurements made by Rogers, Wahrhaftig, and Schomaker in 1947 indicated that it is a pyramidal molecule; the bond angle F—Cl—F is about 86° and the Cl—F distance 1.63 Å. (*15*). Later investigations using infrared and Raman spectra measurements indicate that the molecule is neither planar nor pyramidal, but may be asymmetrical or else strongly associated in the liquid state (*9, 20*).

The value of Trouton's constant for chlorine trifluoride is 20.8, which is very close to the empirical value of 21 for nonassociated liquids with a molecular weight of about 100 (that of ClF_3 being 92.5) and with not too high a boiling point. Thus this evidence would appear to indicate that chlorine trifluoride is not associated in the liquid state, since associated liquids such as water and ethanol generally have larger Trouton constants. However, there is some evidence of partial dimerization in the gaseous state, judging by the extent of deviation from the ideal gas law, and this combined with a similar extent of association in the liquid state would not be inconsistent with a "normal" value for the Trouton constant (*9*).

Further information is meager; no band structures were found in the ultraviolet absorption spectrum, indicating no vibration at these frequencies (*9, 21*). The Raman spectrum shows a line which may be due to an association complex or to a symmetrical pyramid (*22*). Chlorine trifluoride does not form polyhalide salts of the $KClF_4$ type, as contrasted with BrF_3. Thus at the present time, although several electron diffraction, infrared, and Raman spectra studies have been made, no definite conclusions as to the structure of chlorine trifluoride have been reached (*20*).

Bromine trifluoride is a colorless to yellowish-gray liquid, discovered independently in 1905 by P. Lebeau and E. B. R. Prideaux (*9*). It is very reactive, forming the highest known fluorides of the elements from their other halides; it etches glass and quartz; it sets fire to wood and paper and reacts violently with most organic compounds. Bromine trifluoride is insoluble in liquid bromine, floating on its surface. Iodine placed on solid BrF_3 melts with incandescence.

Electron diffraction measurements of BrF_3 indicate that it is pyramidal with the bond angle F—Br—F equal to 86° and the F—Br distance equal to 1.78 Å. (*15*). Specific conductivity measurements decrease over the range 15–60°, following Ohm's law. It was therefore postulated that BrF_3 ionizes to form BrF_2^+ and BrF_4^- ions, the negative temperature coefficient being explained by the thermal instability of the ions. It has a high Trouton constant (25.3), which is compatible with the ionic character. The conductivity of the solid is about one-thousandth that of the liquid, but if there were true electronic conductance, the difference would be much less. Therefore the two ions probably do exist, the stability of one decreasing with increasing temperature. The compound is too reactive to study in solvents, but the existence of the BrF_4^- ion is fairly certain because of such compounds as $KBrF_4$, $Ba(BrF_4)_2$, etc. (*19*). BrF_3 may have an ionic crystal lattice, in which eight BrF_2^+ units surround one of BrF_4^-, analogous to the PCl_4^+-PCl_6^- ionic structure of phosphorus pentachloride (*9, 11*). The BrF_2^+ ion is probably angular rather than linear like the ICl_2^- ion (*9*).

Earlier investigators (*3*) believed BrF_3 to be planar, with the Br atom in the middle of an equilateral triangle and the three F atoms at the three apexes, although they also admitted the possibility of a trigonal pyramid with the Br atom at the apex, *i. e.*, the Br atom

does not lie in the same plane with the F atoms, but at some distance above it. The pyramidal structure agrees with Zachariasen's rule that XY_2 molecules are linear and XY_3 molecules planar when the total number of outer electrons of all sharing atoms equals two times or three times, respectively, the number of electrons in the outer shell of the Y following the inert gases; otherwise a pyramidal structure results (the total number of outer electrons $= 7 + 3 \times 7 = 28$; $3 \times 7 = 21$, but $28 \neq 21$).

Woolf and Emeléus isolated the BrF_2^+ and BrF_4^- ions in 1949 (*24*). The spectrum of BrF_3, measured in 1934 (*25*) is continuous, showing no sign of any vibrational structure. The BrF_4^- ion is similar to the ICl_4^- ion in structure. Since the covalent radii for Br and F are 1.14 Å. and 0.64 Å. respectively, the Br—F distance should be 1.78 Å. and the ion would be smaller than the ICl_4^- ion, in which I—Cl is 2.34 Å. (*26*). Thus most of the recent work indicates that bromine trifluoride exists as the BrF_4^- and BrF_2^+ ions, in which the former is a planar ion with unshared pairs of electrons above and below the plane and perpendicular to it (*14*) and the latter an angular F—Br—F— ion in which the bond angle is not 180° (Figure 4).

Iodine trichloride occurs as lemon-yellow needles, rhombic in shape, and was first prepared by Gay-Lussac and Davy in 1814. It is soluble in most of the ordinary organic solvents and dissolves in water with decomposition. It decomposes almost completely to the monochloride and chlorine in the vapor phase. It obeys Ohm's law when subjected to a direct current voltage (*19*) and is a good conductor in the fused state (*9*), the conductivity (8.4×10^{-3} ohm^{-1} cm.$^{-1}$) being about twice that of the monochloride. The conductivities in the solid and liquid states are almost the same, indicating that there may be true electronic conduction. No decomposition potential has been found.

The structures of the polyhalide ions may indicate the type of structures to be expected in the interhalogen compounds. The ICl_4^- ion contains 12 electrons in the iodine valence shell with unshared electron pairs in the two trans positions of an octahedron (*9, 14*), that is, the ion itself is planar with all four Cl atoms in a square about the central I atom, as previously described in the discussion of the structure of ClF_3. The I—Cl distance is 2.34 Å., as determined by X-ray diffraction analysis of $KICl_4$ (*11*).

ICl_2^+ contains ten electrons in the iodine valence shell, with unshared electron pairs in the equatorial positions of a trigonal bipyramid, so that the structure of the ion itself is linear. Without evidence to the contrary, the ICl_3 molecule is probably planar, with the three chlorine atoms at the corners of an equilateral triangle, the iodine atom being at the center and the two pairs of unshared electrons occupying the apexes of a trigonal bipyramid (Figure 5).

Iodine tribromide is a dark brown liquid, soluble in water and in alcohol. The so-called "iodine tribromide" used medicinally is probably a solution of iodine in bromine in 1:3 molar ratio. However, Faull and Forbes (*27*) found evidence for the existence of IBr_3 as a definite chemical compound. They measured the e. m. f. of a cell consisting of an aqueous iodine-bromine solution containing a platinum-iridium electrode, coupled to a calomel reference electrode, and studied the change as bromine was added, the solution originally containing less than three times as much bromine as iodine. The results indicated that at least part of the bromine was tied up as IBr_3, allowance being made for that definitely bound in the stable compound IBr. From analogy to ICl_3 (in view of the close similarity between Cl and Br), the structure of IBr_2 is probably also that of a planar equilateral triangle, with the two unshared electron pairs occupying positions on either side of the plane of the molecule as in Figure 5.

Two compounds of the general formula AB_5 are known to exist, in which there are five shared electron-pair bonds and one unshared pair of electrons, BrF_5 and IF_5. A third pentahalide, IBr_5, is listed in "The Condensed Chemical Dictionary," 1942 edition (*7*), but it is probably only a mixture of bromine and iodine in a 5:1 ratio.

Bromine pentafluoride is a colorless liquid, first prepared in 1931 by Ruff and Menzel (*9*). It is very reactive, decomposing water with the formation of $BrOF_3$ and HF. Both metals and non-metals ignite when dropped into it, forming the corresponding fluorides.

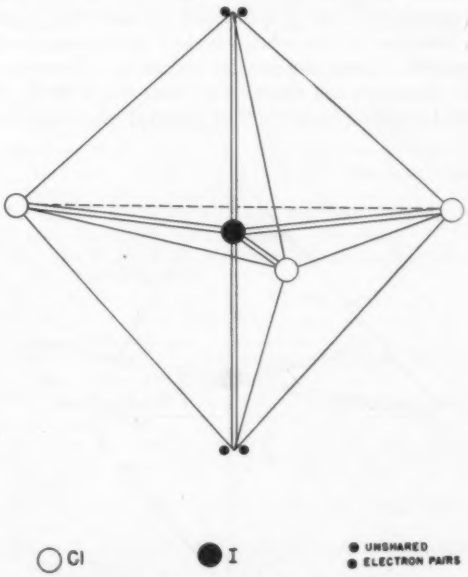

Figure 5. Structure of Iodine Trichloride

It reacts with bromine forming BrF_3 and liberates the free halogens from sodium chloride and potassium bromide (*4*).

Bromine pentafluoride has a rather high Trouton constant, 23.7, indicating that it is at least somewhat associated in the liquid state. Molecular weight determinations indicate that there is no association in the vapor state (*28*). The most probable structure appears

to be a tetragonal pyramid in which the bonds are directed towards the four corners and the apex, each bearing an F atom, with the Br atom inside the pyramid, but probably not in the same plane with the four base fluorines (*14*). The unshared pair of electrons is probably below the plane of the base of the pyramid making an octahedron. Such a structure for BrF_5 is substantiated by comparison with IF_5, whose structure has been fairly well authenticated (*10*)(Figure 6).

Iodine pentafluoride is also a colorless liquid which may have been obtained by Gore in 1871 from the reaction of iodine with silver fluoride at elevated temperatures, but it was not definitely identified and described until 1902 when Moissan prepared it from the elements (*9, 28*). It distills at atmospheric pressure without decomposition. At 500°C. free iodine distills off, and probably iodine heptafluoride. It is somewhat less reactive than the lower fluorides of chlorine and bromine. Most metals are only slightly attacked by it, since a surface coating of fluoride is usually formed. It reacts with arsenic, antimony, and boron, with incandescence, and reacts violently with water and sulfuric acid, but not with nitric acid, hydrogen, or oxygen (*9, 28*).

Considerable work has been done on the structure of iodine pentafluoride in recent years. It shows a continuous absorption spectrum in the visible range, indicating no vibrational structure (*25*). Its electron diffraction pattern indicates that it is not a trigonal bipyramid (*15*) but it is difficult to determine its structure because of the relatively low scattering power of fluorine in X-ray diffraction patterns. However, the I—F distances are much less than the 2.56 Å. which would be appropriate for the trigonal bipyramid structure. A distorted octahedral arrangement of fluorine atoms around a central iodine atom agrees with the X-ray diffraction data if three different I—F distances are used (*15*).

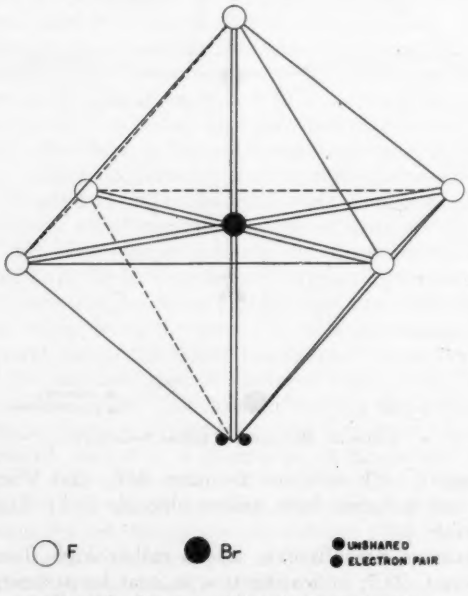

Figure 6. Structure of Bromine Pentafluoride

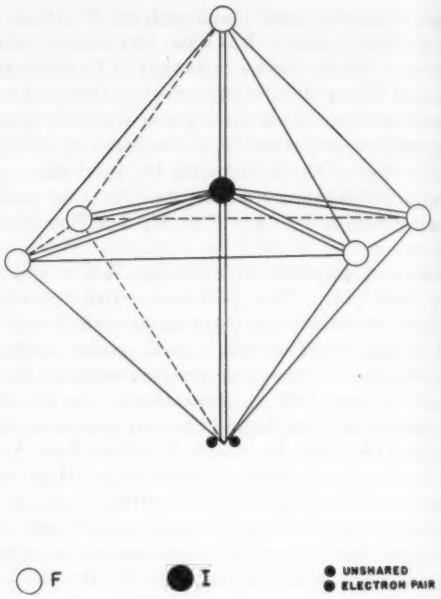

Figure 7. Structure of Iodine Pentafluoride

The specific conductivity is 2–3 $\times$ 10^{-5} $ohm^{-1}cm.^{-1}$ (*19*), indicating some degree of ionization, as is confirmed by the positive temperature coefficient. The existence of IF_6^- ions is also borne out by the formation of compounds such as KIF_6, so that IF_5 may ionize to IF_6^- and IF_4^+ in a manner analogous to that of BrF_5, although probably not to as great an extent.

The most recent work by Lord, Lynch, Schumb, and Slowinski in 1950 (*10*) included both infrared and Raman spectra measurements. Although they encountered several technical difficulties because of the high reactivity of the compound, their measurements were sufficiently accurate to enable them to determine fairly conclusively that the structure should be either a pyramid with four of the F atoms in a square and the I and the fifth atom on the fourfold axis perpendicular to the square (with the F presumably at the apex), or a regular pentagon, in which the five F atoms are on a plane around the central I atom. Although there were some discrepancies between the calculated and observed spectra for both these structures, the data appear to favor the pyramidal structure over the planar structure (Figure 7). This also follows Pauling's pattern for AB_5 molecules with one unshared pair of electrons. The F—I—F angle is 105°; the I—F distance approximately 1.75 Å.

Iodine heptafluoride is unique in that it is the only compound in which a halogen has a valence of seven.

It is a dense snow-white powder or colorless crystals, depending upon the method of preparation and purification. It may be supercooled as a dense, mobile liquid with a sour, musty odor (*28*). It was first prepared by Ruff and Keim in 1930 by fluorination of iodine pentafluoride at 200°C. (*9, 29*). It sublimes at 4°C., forming a colorless gas with a distinctive odor resembling neither fluorine nor iodine. At pressures slightly above atmospheric it melts at 5–6°C. to a colorless liquid.

Iodine heptafluoride decomposes water, acids, and alkalies; it reacts with all metals except those of the platinum group, the rate of reaction depending upon the formation of a fluoride coating. It reacts with iodine to form the pentafluoride, with chlorine to form iodine monochloride, iodine trichloride, and chlorine fluoride, but it apparently does not react with bromine. It reacts rapidly with glass, more slowly with quartz. It liberates bromine from potassium bromide and silver from silver nitrate.

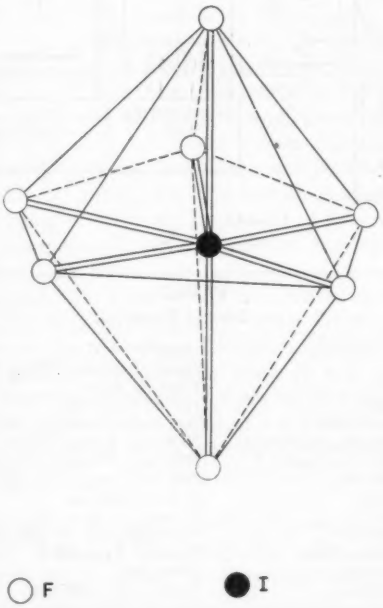

Figure 8. Structure of Iodine Heptafluoride

Iodine heptafluoride is also unique structurally; there is no unshared electron pair, each outer shell electron being shared with a fluorine atom. Lord, Lynch, Schumb, and Slowinski (*10*) investigated the structure of IF_7 and from the combined data obtained from the infrared and Raman spectra concluded that the structure of iodine heptafluoride should be either a plane heptagon or a pentagonal bipyramid, but the discrepancies assuming the latter structure are less than those assuming the former. Thus the IF_7 molecule may be considered to be a pentagonal bipyramid with five fluorine atoms lying in a plane about the central iodine atom and the two remaining fluorine atoms equally spaced above and below this plane to form a pentagonal bipyramid (Figure 8).

ACKNOWLEDGMENT

The author wishes to express her appreciation to Dr. Martin A. Paul of Harpur College for suggesting the subject for this paper, and for his encouragement in preparing it.

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ELECTROLYSIS WITH CONTROLLED CATHODE POTENTIALS[1]

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POTENTIOSTATS, or instruments for the control of the cathode potential during an electrolysis, according to the principles first outlined by Sand,[2] are now becoming so widely known that there seems no need to discuss the theory involved in this paper. So far as the author is aware, however, there has been hitherto no attempt to make use of these instruments in teaching the principles of electroanalysis to undergraduates. The advantages in so doing seem more or less obvious: the chief among them being the ability to separate and analyze an alloy exclusively by electrolytic means, and the direct application to laboratory practice of the theories of electrochemistry.

This paper describes the construction of such a potentiostat and its use in our quantitative analysis laboratory. Because of the limitations of a small-college budget, particular attention was paid to economy and to simplicity of construction and maintenance. Consequently, as much use as possible was made of equipment already on hand and of war surplus materials. The apparatus actually cost just under $100, not including labor or the platinum electrodes. However if we had had to purchase all of the items, it would have cost approximately double this figure, which is still not extreme for an instrument of this type.

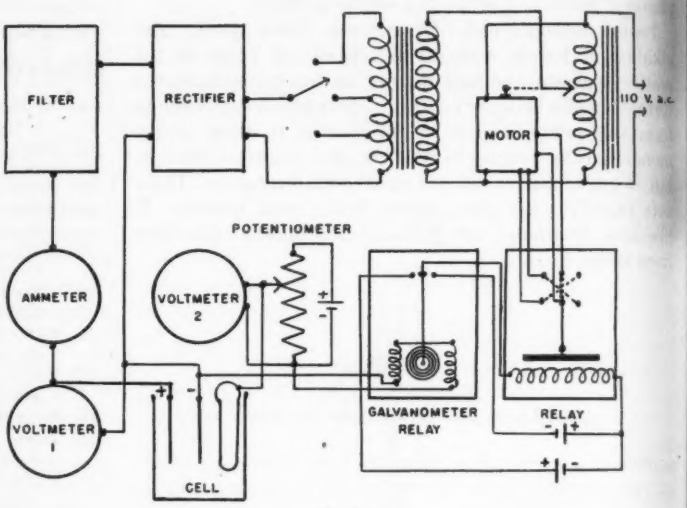

Figure 1

TABLE 1
List of Parts

Pilot lights (2)	Signalite, 110 v.
Motor	Signal universal, Type B 26
Powerstat	Type 20, 60 w.
Transformer	110–24 v., 3 taps
Selenium rectifier	Full-wave, 10 amp., 24 v., a. c.
Electrolytic condensers (2)	3500 mf., 7 amp., 25 v., d. c.
Electrolytic condenser	2000 mf., 7 amp., 50 v., d. c.
Choke coils (2)	0.03 h., 7 amp., 0.2 ohm
Milliammeter	G. E. 0–500 ma.
Voltmeter	Weston, 0–15 v., d. c.
Millivoltmeter	Westinghouse, 0–200 mv., d. c.
Galvanometer relay	Weston, Type 30 H
Polarity relay	2000 ohm
Potentiometer	Radio type, 100 ohm

CONSTRUCTION

Although it was designed and built before his paper appeared, our instrument is essentially the same as Lingane's,[3] except that we employed a polarity relay instead of his two electronic relays, thus eliminating all electronic circuits, and a single variable transformer with friction drive for the control. Figure 1 shows a simplified circuit diagram and Table 1 a list of essential parts with specifications. Of these parts we already had the galvanometer relay, polarity relay, and motor. The other parts were purchased from local radio supply houses, as were, also, the incidential switches, etc., which are not listed.

Figure 2 is a view of the front panel of the completed instrument. Figure 3 shows the electrode holder, with calomel electrode and stirrer. The stirrer was subsequently replaced by a magnetic stirrer, which was found to be more effective and convenient.

Most of the details of construction need not be gone into here. Either they are self-evident from the figures or would in any case be worked out to suit individual need. Two or three items, however, may well be described in greater detail.

The motor which we used to drive the Powerstat was a universal motor equipped with a worm gear. Its final output speed was 50 r. p. m. This was geared to a gear train taken from a Navy range calculator which had a friction drive incorporated into it (actually a mechanical integrator), allowing variation of the speed of the output shaft. The Powerstat was mounted on a

[1] Based on a paper presented before the Division of Chemical Education of the Philadelphia Section of the American Chemical Society, January 18, 1951.

[2] SAND, H. J. S., *J. Chem. Soc.*, 91, 401 (1907).

[3] LINGANE, J. J., *Anal. Chem.*, 22, 1169 (1950).

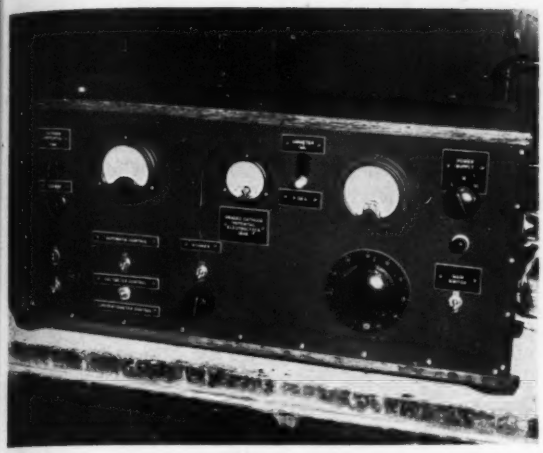

Figure 2

vertical bakelite plate fastened at right angles to the front edge of a brass plate on which was mounted motor and gear train. The wheel at the back of the Powerstat was turned true on the lathe and rested on the output shaft of the gear train, which had a small piece of rubber tubing cemented over it. There was enough spring to the mounting so that the Powerstat could be disengaged from the train by pulling outward and downward on its knob, and could thus be set by hand. Adjustment of the train was made so that the speed of the Powerstat was less that 1/4 r. p. m.

A number of other motors and gear trains are on the market which will serve equally well. We have also used, for example, a General Electric Inductor Motor, Model 5SMY20J, with its gear train (6 r. p. m.), by making a slight modification. A geared Holtzer-Cabot motor, RWC 2505, with a speed of 1 r. p. m. is also on the market, and could be made to serve by supplying its output shaft with a rubber-covered pulley of suitable size.

The milliammeter was provided with a shunt made of number 14 stranded wire adjusted after assembly against a precision ammeter so that a reading of 1 milliampere on the scale equaled 0.02 ampere. A heavy knife switch with its handle protruding through a slot in the panel enabled this shunt to be cut in or out. The meter could thus read from 0 to 500 milliamperes or from 0 to 10 amperes.

The polarity relay was originally provided with two contacts and remained closed on the side of the last operation, under the influence of the permanent magnet. Two sets of spring contacts from a telephone relay were added to this so that it acted as a double-pole, double-throw relay, normally open. When connected as shown in Figure 1, it followed the action of the galvanometer relay and started or stopped the motor in either direction.

Voltmeter 2 (0–200 millivolts) was carefully calibrated against a good potentiometer and adjusted by series resistances to read volts, and until it was accurate to the nearest 0.02 volt. The scale was changed by the introduction of decimal points to read 0–2 volts.

OPERATING CHARACTERISTICS

The effectiveness of the rectifier and filter circuit was tested by connecting an oscilloscope to the output, with a large carbon rheostat as a load. The oscilloscope trace was a perfectly straight line at all loads up to 10 amperes.

The response of the instrument was tested by running several electrolyses for copper with a recording galvanometer connected into the calomel cell circuit. The records obtained showed a maximum variation of about 15 to 20 millivolts.

A number of actual analyses were run before the apparatus was put in student hands. Table 2 shows the analysis for silver of a solution containing copper and silver in the approximate proportions of coin silver. The silver was deposited out first from an ammoniacal solution at a potential of −0.10 volt versus the S. C. E. at the beginning of the electrolysis and at −0.20 volt toward the end. Copper was determined on a conven-

TABLE 2

	Wt. Ag found, g. (0.2250 g. Ag present)
1	0.2252
2	0.2249
3	0.2247
4	0.2251
5	0.2249
6	0.2244
7	0.2246
8	0.2247
9	0.2247
Mean	0.2248

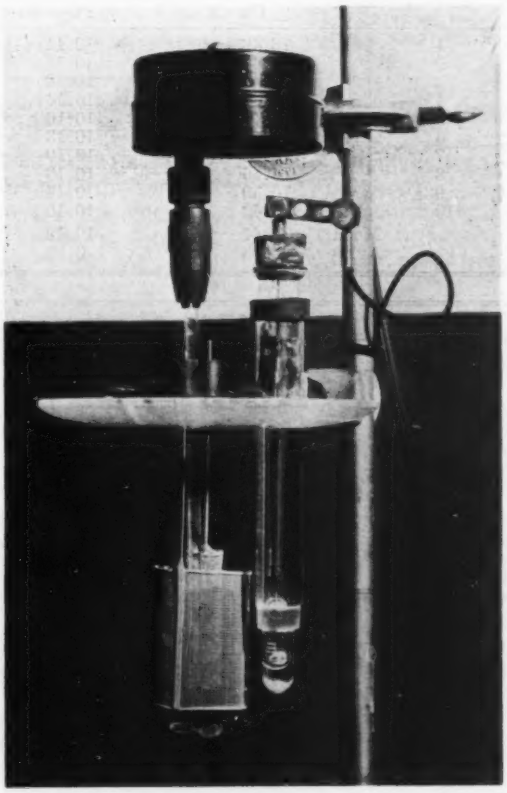

Figure 3

TABLE 3

	% Ag	% Cu	Sum
1	89.95	9.90	99.85
2	89.95	9.98	99.93
3	90.10	10.14	100.24
4	89.90	10.10	100.00
5	90.02	10.10	100.12
6	90.10	10.07	100.17
7	90.01	10.08	100.09
8	89.91	10.18	100.09
Mean	89.99	10.07	100.06
Av. dev.	0.07	0.06	

tional apparatus without control, and the results are not shown.

Table 3 shows the results of a series of analyses of an actual silver coin done in the same way. The copper results are included here, since the composition of the metal was not known, so that the only check on accuracy was the sum of the two determinations. In Nos. 1 and 2 the potential was not raised at the end of the silver determination.

Table 4 gives the results of a series of analyses of a brass for copper and for tin and lead combined. The copper was deposited from chloride solution at a controlled potential of −0.40 volt, and tin and lead from the resulting solution at a potential of −0.70 volt, following the directions of Diehl.[4] Zinc was also determined, but by conventional methods, and the results are not included in the table. The sample was Thorn Smith's No. 321, and was reported to contain 76.39 per cent copper and 10.13 per cent combined tin and lead. Separation of the tin and lead was not attempted, as this was considered a rather difficult analysis for student use.

TABLE 4

	% Cu	% Pb + Sn
1	76.48	10.11
2	76.45	10.14
3	76.49	10.10
4	76.38	10.14
5	76.44	10.10
6	76.42	10.13
7	76.48	10.10
8	76.46	10.13
9	76.39	10.14
10	76.42	10.10
Mean	76.44	10.12
Av. dev.	0.03	0.02

TABLE 5

	1	2	3	4	5	6
Copper in Brass						
Student mean	78.05	76.46	87.56	82.31	84.09	82.09
Theoretical	78.01	76.43	87.64	82.03	84.11	82.03
Av. dev.	0.04	0.03	0.08	0.28	0.21	0.06
Parts/1000	0.5	0.4	0.9	3.0	2.5	0.7
Tin and Lead in Brass						
Student mean	10.02	10.10	10.55	9.82	9.93	9.96
Theoretical	9.93	10.12	10.54	9.90	9.93	9.90
Av. dev.	0.09	0.02	0.01	0.08	0.00	0.06
Parts/1000	9.0	2.0	1.0	8.0	0.0	6.0

STUDENT RESULTS

During the fall of 1950 a group of six students in the author's laboratory used the instrument for the analysis of bronzes for copper and for tin and lead combined. The conventional method for bronze involves the gravimetric determination of tin as oxide, lead as sulfate, followed by electrolysis of copper, then gravimetric determination of zinc as pyrophosphate. Controlled cathode methods allow the complete electrolytic analysis of the bronze: first copper from chloride solution at 0.40 volt in the presence of hydroxylamine hydrochloride to prevent anodic oxidation of the platinum. Tin and lead are then deposited together at a higher potential (0.7 volt). They can be separated by dissolution in nitric and hydrofluoric acids, followed by anodic deposition of the lead as PbO_2. Zinc may be deposited at constant amperage from the copper filtrate, in ammonia solution.

Because of time limitations only copper and combined tin and lead were determined. Table 5 shows the results of these analyses.

In addition to using the apparatus for analyses involving separations, we have also found it convenient in some cases where separations are not necessary, for example, the analysis of copper oxide ores. The advantage lies in the fact that electrolysis in a chloride solution is much more rapid as a rule than in a sulfate-nitrate solution and that the end of the analysis is marked by the low and nearly constant current.

The instrument can, of course, be used for electrolyses without potential control, by simply leaving the control switches turned off, or for a source of direct current for any purpose such as charging storage batteries. The rectifier and transformer used were rated at 10 amperes, the chokes and condensers only at seven, but such apparatus is usually conventionally rated, and we have drawn 10 amperes from it for considerable periods without damage.

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QUANTITATIVE PAPER CHROMATOGRAPHY FOR STUDENTS

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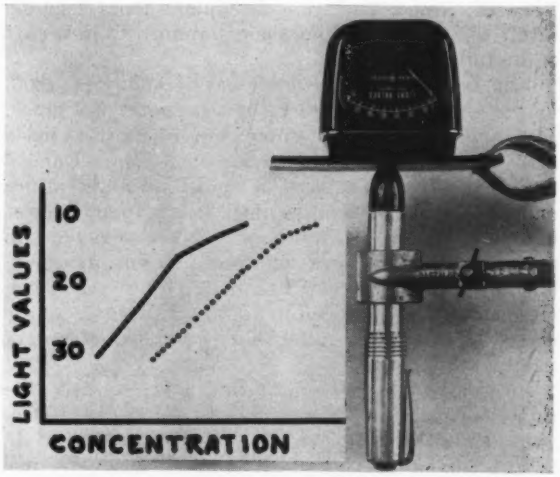
After the student has completed qualitative experiments,[1] he is ready to learn the elementary principles of quantitative paper chromatography. This involves measurement of spot density with a densitometer, and is based upon the observation that, within limits, the maximum color density of each spot is proportional to the concentration of material in the entire spot.[2] To demonstrate this relationship, and practice the basic procedure, the student may well begin with a standard curve for alanine.

Prepare 10 ml. of 0.1 *M* solution by dissolving 89.1 mg. of alanine in 10 per cent isopropanol (preservative). Remove 5 ml. and dilute to 10 ml. producing a 0.05 *M* solution. Continue serial dilution in this way to yield 0.025, 0.0125, and 0.00625 *M* solutions. Mark a pencil line 2 cm. from a long edge of filter paper 15 × 22 cm. The paper should be optically uniform; several types are suitable, including Whatman No. 1. Apply a 2-μl. spot of each molarity, 2.5 cm. apart and just above the pencil line. The spots can be applied with a self-filling transfer-type pipet capillary (Microchemical Specialties Co., 1834 University Ave., Berkeley 3, California). Allow the spots to dry, staple the sheet to form a cylinder, and chromatograph about 3 hours in a closed 1-quart, wide-mouthed, Mason jar containing 1 cm. of 80 per cent reagent grade phenol in the bottom. A room temperature near 70°F. is preferable; while chromatographing, avoid sudden temperature fluctuation and exposure to bright light or radiant heat.

Remove the papergram, unstaple, and hang 24 hours at room temperature (preferably in a hood) to remove phenol. Spray uniformly with 0.2 per cent ninhydrin in 95 per cent ethanol. Hang in a dark place at room temperature for 18 hours. After this time the spots have reached maximum density and can be measured in a densitometer. An expensive instrument is not necessary for this experiment. The accompanying figure shows a satisfactory arrangement, consisting of a small flashlight and a photographic exposure meter. The prefocused bulb is inserted into a 5-mm. hole punched in black cardboard. With the light on, the densest portion of each spot is placed over the aperture. The exposure meter is placed over the paper, and the light value reading is recorded.

These values, plotted against concentration, give a standard curve for alanine. Replications agree very closely. A standard curve for alanine obtained in this way is shown by the solid line in the figure. The dotted line shows the curve obtained with a more elaborate densitometer, the Densichron, and is included for purposes of comparison.



Once the standard curve is obtained, it can be used to compute the concentration of an unknown alanine solution chromatographed under the same conditions. It is necessary, of course, that the concentration of the unknown solution be adjusted to fall within the range of the standard curve. Further experiments, including both identification and quantitative determination of "unknowns" by paper chromatography, will readily suggest themselves to the instructor. This principle of analysis is applicable to practically all of the amino acids, and with suitable modifications to any substance which can be separated on a papergram and for which a suitable color reagent is available.

The above procedure is not presented as the ultimate in refined analytical procedure. Obviously for careful analytical work better instrumentation and many refinements of technique are essential. Quantitative paper chromatography shows great promise with regard to accuracy, precision, versatility, ease, and rapidity. The limits of this new procedure have not yet been completely defined. For more advanced work the author recommends use of a Densichron with a paper guide, equilibration by means of reference spots, and measurement of increment curve areas with a polar planimeter.[3]

[1] Patton, A. R., J. Chem. Educ., **27**, 60, 574 (1950).
[2] Block, R. J., *Anal. Chem.*, **22**, 1327 (1950).
[3] Patton, A. R., and Patricia Chism, *Anal. Chem.*, in press.

JEAN BAPTISTE ANDRE DUMAS

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In the year 1823 the young J. B. Dumas came to Paris, his eyes filled with the glamorous city of the Restoration, and his mind filled with the Baron von Humboldt's description of the great men of science who lived and worked there, Gay-Lussac, Ampère, Thenard, Arago, Laplace. To Dumas Paris held the future for science, and he was determined to be a part of this future.

Jean Baptiste André Dumas was born July 14, 1800, in the small town of Alais in the south of France. Alais was an industrial center close to the coal fields and the iron, antimony, and lead mines. Dumas' father was the town clerk, a man fond of literature and art, who may have inspired his son to an interest in study. From his youth Dumas showed a desire for adventure. He first determined to join the navy, but the troubles of 1814–15 prevented him. And then, after a short apprenticeship to an apothecary, at the age of 16 he set out on foot for Geneva, the home of relatives.

In Geneva Dumas continued under LeRoyer the subject of pharmacy which he had begun in his native village. But soon, under the influence of Gaspard de la Rive, he became interested in chemistry. His first investigation was on the equivalent amounts of water in the sulfates and other salts of commerce, and this work was highly commended by de la Rive. He then turned to work on atomic volumes and densities and became interested in compound ethers. Dumas taught his first class in chemistry at this time to a group of pharmacy students, and the first appearance of Dumas' name in a journal occurred during his stay in Geneva. It was in a paper by Dr. Coindet on the application of iodine to the treatment of goiter, for which Dumas had done the analytical work. Later Dumas turned to physiology and published many papers with Dr. Prevost on the nature of blood, the presence of urea in the body, muscular contraction, etc.

It was at Geneva that Dumas met the interesting Baron von Humboldt, the famous German naturalist and traveler. Attracted to Dumas by his papers on blood, von Humboldt visited him in 1822, and Dumas listened with wonder to his tales of science in the French capital, "of the happy collaboration of men of science and of the unlimited facilities which the French capital offered to young men wishing to devote themselves to scientific pursuits."[1] Dumas determined to go to Paris as soon as possible, but he never forgot his debt to the Geneva academy where, he said, it was proved "that the nobility of science is inherited the same as that of blood."[2]

In Paris Dumas was not disappointed in his expectations. He made many friends among the young men of science studying there: H. Milne Edwards, the physiologist; Adolphe Brongniart, the botanist; and Liebig, who was completing work on the fulminates with the assistance of Gay-Lussac. Dumas became the favorite pupil of Thenard, and his first paper given at the Academy of Sciences was followed by an invitation to dinner by the noted astronomer, Laplace. Through the influence of Arago and Ampère, he was appointed Répêtiteur de Chimie under Thenard at the École Polytechnique and professor of chemistry at the Athenaeum, where evening lectures on literature and science were offered.

Dumas' first years in Paris were occupied not only with his teaching and his experimental investigations, but also with literary work, which included the first volume of his book, "Traité de chimie appliquée aux arts,"[3] and the founding of a journal, the *Annales des sciences naturelles*, with Adolphe Brongniart, the young biologist, and his brother-in-law, Victor Audoin. In 1826 Dumas joined the family by marrying Herminie Brongniart, Adolphe's sister and the daughter of Alexandre Brongniart. Alexandre Brongniart was an important figure in Paris at this time. A celebrated geologist and zoologist, he was also director of the porcelain works at Sèvres. Dumas' marriage was an important step in his rise to recognition.

In the first two decades of the period of his activity in chemistry Dumas' experimental interests were varied and numerous. With Boullay he investigated the compound ethers, defining the relationship between alcohol and its derivatives and developing the first theory in organic chemistry of the constitution of a group of related compounds, the etherin theory. In this theory he compared ammonia and its compounds to certain organic compounds which he considered to be formed from olefiant gas, C_2H_4 (named the etherin radical by Berzelius). According to Dumas alcohol, $C_2H_4.H_2O$, was comparable to ammonium hydroxide, $NH_3.H_2O$, ethyl chloride to ammonium chloride, etc. This investigation led him to the discovery of oxamide and other amides and amic acids.

[1] Hofmann, A. W., *Nature*, **21**, extra no., Feb. 6, p. vii (1880). Hofmann's biography is interesting reading, but not always reliable as to Dumas' contributions to the development of chemistry.

[2] Browne, C. A., "Banquet des chimistes," J. Chem. Educ., **15**, 254 (1938).

[3] This book was published in eight volumes between 1828 and 1848.

With Péligot Dumas isolated methyl alcohol from wood distillate, purified it, prepared many of its derivatives, and recognized its similarity to ethyl alcohol. Later he isolated and identified as alcohols compounds from spermaceti and potatoes, thereby establishing a general series of alcohols. In 1843, in the *Annales de chimie et de physique*, he suggested a homologous series of fatty acids and predicted the discovery of then unknown acids which would complete the series. In this paper he clearly states that the difference between these acids is the C_2H_4 (CH_2) group. The paper was first published in *Comptes rendus* in November, 1842, only a few months after Schiel's publication of a very limited homologous series of alcohols.[4] It is interesting to note, however, that Schiel included glycerol in his series, which may indicate that he did not fully understand the principle of homology. Dumas' clear exposition of homology compared with Schiel's confused one points to Dumas, not Schiel, as the "Father of Homology."

Dumas' most famous work in the field of organic chemistry was that done on substitution. Gay-Lussac made the first observation of this phenomenon in regard to the bleaching action of chlorine. He said that chlorine forms hydrochloric acid from part of the hydrogen of oils "and at the same time part of the chlorine combines with the oil and takes the place (*prend la place*) of the hydrogen removed."[5]

Dumas' interest in substitution, however, sprang from an incident at the royal court in the palace of the Tuileries. The guests at a soirée were extremely annoyed by irritating fumes which came from the burning candles. As director of Sèvres and, therefore, court chemist, Alexandre Brongniart was assigned the task of explaining the phenomenon. He turned the problem over to his son-in-law, Dumas, who found that the fumes were hydrogen chloride released as the candles burned.[6] He found that the chlorine had not been absorbed as such in the bleaching process, but was substituted in the wax. This phenomenon he called metalepsy (exchange) and devised some empirical rules of substitution. A continuation of this investigation of chlorine in organic compounds resulted in his discovery of the correct formulas of chloral and chloroform. It was Dumas who suggested to Laurent that he chlorinate naphthalene, an investigation which eventually led to the true understanding of substitution. In 1838 Dumas' synthesis of trichloroacetic acid proved conclusively, even to the faithful adherents of the electrochemical theory of Berzelius, that chlorine could be substituted in a carbon compound without materially changing its fundamental properties. But, with the enthusiasm characteristic of Dumas whenever he had proved an idea experimentally, he went too far in his speculations on substitution, and, as a result, there arose the polemics on the subject issued in the current journals. The French school, for the most part, upheld Dumas, while Liebig and his followers took a more conservative view, supporting the electrochemical theory and Berzelius.

Dumas' later work in organic chemistry included the preparation of the nitriles from the ammonium salts and their hydrolysis, the preparation of the organic nitrates, and the characterization of camphor and artificial camphor.

In the field of analytical chemistry Dumas developed his vapor density method to determine molecular weights. Gay-Lussac had originated this idea of the importance of vapor density in 1815. Dumas' development of this procedure was simple and accurate.[7] Dumas also developed a method of nitrogen analysis

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for organic compounds, which was based on Gay-Lussac's combustion method.[8] With Stas, Dumas accurately determined the atomic weights of hydrogen, oxygen, carbon, and many other elements. He also investigated the composition of air and of water. All this work was done before 1845.

When the Second Republic was formed in 1848, Dumas became very active in the affairs of his country. He was elected to the National Assembly in 1848 and served as Minister of Agriculture and Commerce and in 1868 as Master of the Mint. He became a member of the municipal council of Paris in 1854 and soon rose to its presidency. During his administra-

[4] Neither Schiel nor Dumas used the word homology. Gerhardt adopted the idea in part in 1844 and coined the word.

[5] PARTINGTON, J. R., "A Short History of Chemistry," Macmillan and Co., London, **1948**, p. 240.

[6] HOFMANN, A. W., *loc. cit.*, p. xiv.

[7] DUMAS, J. B. A., "Mémoire sur quelques points de la théorie atomistique," *Ann. chim. phys.*, [2] **33**, 341 (1826).

[8] DUMAS, J. B. A., "Sur l'oxamide," *ibid.*, [2] **44**, 133 (1830).

tion many improvements were made in the drainage and lighting systems of Paris, and the scheme of supplying the city with spring water by aqueducts and tunnels was instigated. After the political upheaval of 1870 Dumas returned to chemistry and to the service of science. He had been a member of the Academy of Sciences for many years and was also a member of the French Academy. He headed many committees, such as the international permanent commission on the meter. He remained active almost until his death, publishing a paper on fermentation in 1872 and one on the occlusion of oxygen on silver in 1878. Dumas received many honors during his lifetime, among which were the Faraday medal, the Copley medal, and the Grand Cross of the Legion of Honor. Dumas died in Cannes in 1884.

Dumas was a man of many talents and varied interests. Not only was he renowned for his careful analytical work, much of which has not been changed today, but he also was interested in chemical philosophy. He was a prolific and excellent writer and lecturer. His prose is clear and concise, but it is written with such ease, intelligence, and enthusiasm that it becomes interesting as well as informative reading.

Dumas was also a great teacher and lecturer. He was professor of chemistry at the École Polytechnique, the École de Médecine, and the Sorbonne, as well as the École Centrale. His classes, Pasteur says, were always filled long before the hour,[9] and his lectures were full of interesting facts, anecdotes, and demonstrations. Yet he never lost sight of the subject, and his lectures were masterpieces of the integration of ideas. Dumas also insisted upon the importance of laboratory teaching. He was the first to introduce into France the fine laboratory teaching that Liebig had begun at Giessen. As early as 1832 he founded a laboratory at his own expense for his students. He furthered the education of scientists in France by the founding with T. Olivier and E. Pictet of the École Centrale des Arts et Manufactures for the study of chemical engineering. He also organized the Society for the Encouragement of the Sciences. Dumas was the inspiration and guide for many young chemists, both from France and the surrounding countries. Piria, Stas, Melsens, Wurtz, Laurent, and Pasteur all studied under the "Master."

The character of Dumas was full of contradictions. Primarily, he was a conservative; in religion, he was orthodox;[10] in politics, he followed the middle road. He was slow to accept a theory. Over and over in the "Leçons de philosophie chimique" he insists that experimentation is the first law of chemistry. He insists on "blind submission to the power of facts." "Ancient or modern," he says, "chemists see with the eyes of the body before using those of the mind: they make theories to suit facts, and not seek facts for preconceived theories."[11] Only after he himself chlorinated acetic acid did Dumas acknowledge the correctness of the assumptions of Laurent's theory of substitution, although the theory had been suggested in his own empirical rules. Yet Dumas often extended a theory beyond its reasonable limits. After accepting the substitution theory of Laurent and developing it in opposition to the dualistic formulation of Berzelius, he went so far as to suggest that sulfur might replace carbon in an organic compound with little change in the fundamental nature and properties of the original substance.

During the Restoration, when the ideas of the divine right of kings and the power of authority were in vogue, the Academy assumed the regulation of the theoretical speculations of all scientists. The conservatism of Dumas in this early period in chemistry was probably the result of the opposition that he met from Gay-Lussac and Thenard when he first began to work in Paris. Liebig, in a letter to Gerhardt, remarked that nothing injured Dumas more in his early years in Paris than his tendency to theorize.[12] Thus Dumas seems to have adopted the ways of Parisian society and to have accepted the more conservative ideas of the Academy. He himself was practically the dictator of French chemistry from 1840 until about 1865, when Berthelot rose to prominence. Dumas was largely responsible for the unforgivable exiling of Laurent, Gerhardt, and others to the provincial universities.[13] Especially did Dumas fear Laurent and Gerhardt. Laurent had humiliated Dumas in his papers claiming priority to the theory of types,[14] and Dumas could never forgive him. Ostwald, in his life of Gerhardt, has suggested that Dumas also recognized in Gerhardt a man of fiery and unyielding disposition, who would have become a dictator had he gained power in the field of chemistry.

Dumas was very ambitious. He was always careful to meet and know the right people. He married into a fine family. He altered his ideas to suit those around him. It has been said that Dumas borrowed liberally the ideas of other chemists and offered them as his own. Berzelius, in a letter to Wöhler, called him a "charlatan, French wind-bag, chemical dancing-master."[15] Liebig, Laurent, and Kolbe all accused him of violating their priorities. Most certainly Dumas was lax in acknowledging sources. But this was a period of strong words and accusations, and authorities dis-

[9] DUBOS, RENÉ, "Louis Pasteur," Little Brown, Boston, 1950, p. 31.

[10] "Littre, Dumas, Pasteur, and Taine," *Edinburgh Review*, 156, 105 (1882).

[11] DUMAS, J. B. A., "Leçons de philosophie chimique," Gauthier-Villars, Paris, 1937, p. 3.

[12] OSTWALD, W., "Grosse Männer," Akademische Verlagsgesellschaft, Leipzig, 1910, p. 227.

[13] DAUMAS, M., "L'école des chimistes Français vers 1840," *Chymia*, 1, 63 (1948).

[14] Dumas published his research on indigo under the title, "Fourth memoir on chemical types," and Laurent then published his next paper under the title, "Twentieth memoir on types or derived radicals, types which were not discovered by M. Dumas." GRIMAUX, É. "Auguste Laurent," *Rev. Sci.*, [4] 6, 168 (1896).

[15] VON MEYER, E. A., "A History of Chemistry," Macmillan and Co., London, 1906, p. 285.

agree on the truth of these statements. It seems strange that so dishonest a man, as Dumas is made to seem, could gain the world-wide acclaim which he enjoyed. In speaking of the Brongniarts Dumas said, "Animated by the love of truth and indifferent to the enjoyments of fortune, they have found their reward in public esteem."[16] Perhaps this reward was what Dumas was ever seeking.

Dumas seemed to understand most of the problems and theories of his day, yet he was slow to accept them. Whether this was because he found them hard to express or because of his insistence on visible experimental proof is difficult to say. Dumas seemed to grasp the phenomenon of substitution and gave a set of rules to cover it, but left its expression and development to Laurent. He foresaw the principle of homology after his discovery of amyl alcohol. He recognized the classes of compounds in his type theory. As early as 1826 Dumas accepted the truth of Avogadro's hypothesis and recognized the difference between the physicist's atom (molecule) and the chemist's atom (atom), yet he said at one time that he wished the word atom had never been invented. He pioneered the work in atomic volumes. He made his careful determinations of atomic weights in order to prove Prout's hypothesis and found that the atoms were multiples of $^1/_2$ and $^1/_4$ Prout's value. He recognized the classification of the metals into families (triads). However, with the exception of the etherin theory, a work of his early years, he was never the first to publish a theory or to accept it.

The personality of Dumas must have been very engaging. He made friends wherever he went. All the scientific world acclaimed him. At the Congress of Karlsruhe the whole assembly greeted his entrance into an afternoon meeting with an ovation. Wurtz called him the founder of organic chemistry. Liebig dedicated his volume of chemical letters to Dumas. In his personal relations Dumas was kind, genial, and concerned with the welfare of others. As Minister of Agriculture and Commerce he introduced bill after bill to improve the condition of the working class and the aged. Louis Napoleon called him the "poet of hygiene."[17] Dumas was always interested in improving the agriculture and industries of France. He was active and enthusiastic in all that he did, always ready to take advantage of an opportunity. For instance, he found a method of manufacturing sulfates by noticing their accumulation on a porous curtain in a bath house filled with hydrogen sulfide. Dumas attacked each problem with vigor and intelligence and usually succeeded in his endeavors.

Whether as "French wind-bag," "poet of hygiene," or "Master," Dumas must always be remembered as a man who made a great impression on the world of his day in politics, literature, philosophy, and science. His dream of becoming a part of the history of science was fully realized and fully deserved.

[16] HOFMANN, A. W., *loc. cit.*, p. xl.

[17] COOKE, J. P., "Jean Baptiste André Dumas," *Am. J. Sci.*, [3] **28**, 299 (1884).

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ON ACCENTING OBSERVATIONS IN CHEMISTRY[1]

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STUDENTS often remember the equation for the reaction of copper with nitric acid in terms of the magic ratio of 3 to 8. Similar feats of memory pay off on many another chemical question and laboratory experiment. Progress in a science is, of course, impossible without memory but it may be advisable to memorize somewhat more fundamental ideas than the coefficients in an equation. If such memory were correlated with direct experimental observation the situation would be much closer to ideal.

In the writing of chemical equations it has become customary to learn formulas of reagents and products and to balance the stoichiometry first and then to indicate physical states, colors, and odors. A much more firmly gounded scientific approach would be to indicate first any changes in color or any disappearance or formation of phases and then to identify these direct observations with the chemical species causing them. The copper-nitric acid equation would start out in the form:

$$\underset{\text{red}}{Cu} + \underset{\text{conc.}}{H^+} + NO_3^- \rightarrow \underset{\text{dark brown gas}}{\quad} + \underset{\text{blue solution}}{\quad}$$

The dark brown gas would next be identified as NO_2 and the blue color as caused by the presence of Cu^{++} in solution. The stoichiometry could then follow naturally from the direct experiment rather than experimental confirmation following from predicted stoichiometry.

Sight is by far the most used of the five physical senses in the study of chemistry; smell and taste must be applied with great discrimination around a laboratory. Hearing (except for dodging explosions) is of minimal importance, and feel is limited ordinarily to superficial identification. Yet it is doubtful that many students realize the importance of their fundamental sight perception and understand the basis of their chemical conclusions in terms of the type of sight observations made. The evidence for the insolubility of silver chloride is not a set of solubility rules in a text but the observation of a white precipitate when solutions of silver nitrate and sodium chloride are mixed, coupled with the fact that sodium nitrate does not precipitate under these conditions as a glance at the side shelf reagent bottle will show.

One of the basic objectives of a beginning chemistry course is to teach the values of accurate observation. Ability to make such accurate direct observations and couple them with memory to give appropriate deductions is a more valuable trait than memory alone. Rather than phrase questions such as "write the equation for the reaction of copper with concentrated nitric acid" it proves more rewarding to request "the equation for the process in which a brown gas is evolved when copper is treated with nitric acid." Better yet for teaching purposes is the performance of the experiment in which copper is added to the nitric acid, the student being told to observe the changes and write equations describing them. No "key words," such as "brown gas," are supplied. He is on his own. Such testing checks on the student's ability to observe, to record, and to deduce from direct observation, rather than merely to recall certain facts associated with a standard set of familiar words.

Use of this direct demonstration type of question on examinations has a startling ability to distinguish between the student who merely reads the book and remembers the words, and the student who is really observing and making deductions on the basis of those observations.

In actual practice the experiments performed on examinations may not be those which have already been thoroughly discussed but, rather, some which are new in their detail while embodying previously observed processes. These variations are limited only by the ingenuity of the teacher.

Much of the value of this type of teaching lies in the lack of the "key words" by which many students learn. These words are not supplied by the teacher; rather the observations must be made by the student himself. "Azure blue," for instance, on examinations has come to mean the presence of copper ammonia complex ion almost universally, just as "pungent" indicates the presence of chlorine. It is surprising how many students will fail to recognize the substance or its formula when an experiment is done in which the azure blue appears without attention being called specifically to it.

A chemical equation is, for many a student, such a complete abstraction that he would be hard put to it to describe the actual observations which would be made in a process for which he was supplied the complete equation. Such abstractions may be more and more necessary and feasible as the training of a chemist proceeds, but it would seem that the experimental basis for them might well deserve greater accent, particularly with the beginning student.

We have now been using demonstration examinations for five years and find that the students actually enjoy taking them compared to the usual type. The best students on the other exams are the best students on these; the worst students are still the worst. But in the middle of the class, and just below the top level, it is

[1] Presented at the 119th Meeting of the American Chemical Society, Boston, April, 1951.

quite common to find students who do considerably poorer work on an observational exam. These are the ones who depend too much on remembered words and phrases; who are too inarticulate to phrase their own observations understandably. And there are others who have used their eyes throughout the course, and, hence, do well on these exams. It seems clear that such exams test another facet of the student, not previously measured, and so further round out, and improve, the picture of his accomplishments with respect to the class as a whole.

It is important to train students to be careful readers, as in the usual test, and to be good listeners, as in the oral test, but it seems equally desirable to encourage them to be good direct observers, as in the demonstration test.

COULOMB'S LAW AND THE QUALITATIVE INTERPRETATION OF REACTION RATES

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THE following discussion is pertinent only when electrostatic influences upon ions in solution predominate over specific effects such as those arising from the solvent due to solvation (*1*) and other causes (*2*).

Coulomb's law for ions in solution may be written for high dilution:

$$F = \frac{Z_1 \epsilon \, Z_2 \epsilon}{Dr^2} \qquad (1)$$

where F is the force between two ions of charges $Z_1\epsilon$ and $Z_2\epsilon$ respectively, when the two ions are separated by a distance r in a medium of dielectric constant D. This dielectric constant D can for all practical purposes, especially in dilute solutions, be identified with that of the pure solvent. The Z's represent the valencies of the ions and ϵ is the electronic charge.

The force F is attractive for ions of unlike sign but is repulsive for ions of like sign.

Before we really use Coulomb's law for a qualitative interpretation of kinetic data we should have clearly in mind the Debye-Hückel interionic attraction theory. The theory pictures an ion as being on a time average surrounded in a spherically symmetrical fashion by an atmosphere of ions of opposite charge to that of the central ion. This atmosphere would partially neutralize the charge of the central ion with respect to its effect upon some ion outside the atmosphere.

Now let us see what Coulomb's law would tell us about the effect upon ionic reaction rates of increasing the ion concentration of the solution at constant dielectric constant and constant temperature. As ion concentration increases, the ion atmospheres surrounding any particular species of ion will become more dense. On the other hand, at infinite dilution there would be no atmospheres surrounding any species of ion. Consider reactants of unlike charge so that the Coulomb force is an attractive one. The attractive force between the reactant ions at high dilution would be given by equation (1). Now increase the ionic concentration by dissolving dissimilar ions in the solution. The reactant ions would begin to form atmospheres the density of which would depend on the total ion concentration in the solution. But when the atmosphere forms, the effective charge on the ion is lessened with respect to its influence on other ions. Thus the oppositely charged reactants will attract each other less strongly depending on the ion concentration in the solution. This can be seen from equation (1), for when $Z_1\epsilon$ and $Z_2\epsilon$ are diminished, the force F which is attractive in the case being considered becomes smaller at constant D and r. Thus, from Coulomb's law, it can be predicted that when the total ion concentration is increased in a solution containing reactants of unlike sign, the attractive forces between the reactants will be lessened and their rate of reaction decreased. This result can be experimentally verified and quantitatively calculated using the Brønsted-Debye equation (*3*).

In the case of reactants of like charge the ion atmosphere effect will again be a decrease in the force between the reactants, but in this case the force is repulsive, and by decreasing the repulsive force the ability

of the reactants to contact each other is enhanced and their rate of reaction increased. The amount of increase will be a function of the amount of cancellation of charge, and therefore a function of the atmospheric density, and hence of the total ion concentration of the solution. Thus equation (1) would predict a lessening of repulsive forces with decreasing effective charge due to ion atmosphere effects, and therefore would predict an increasing rate of reaction between reactants of like sign with increasing total ion concentration. This prediction can also be experimentally verified and the result can be calculated from the Brønsted-Debye theory (*3*).

Consider the effect upon the rate of ionic reactions of changing the dielectric constant of the solvent at constant ionic strength and constant temperature. From Coulomb's law, if D is increased, other factors remaining constant, the force between ions becomes smaller. For ions of unlike sign the force is attractive and, as D increases, the attractive force decreases and the ionic reactants get together less readily; hence, the rate of reaction between such reactants would be decreased by raising the dielectric constant of the solvent.

For ions of like sign the increased dielectric constant would cause a decrease in repulsive force and therefore ionic reactants of like sign would contact each other more readily and their rate of reaction would consequently be increased.

When the dielectric constant of the medium is decreased, the rates of reaction between ions of unlike sign would be increased due to increased attractive forces among the particles. For like charge of ionic reactants the rate of reaction would be reduced by decreasing the dielectric constant of the medium since the repulsive forces among the particles would be enhanced.

All these effects have been experimentally verified (*2, 4, 5*).

The question might arise as to how the dielectric constant of a solvent can be controlled at constant temperature. The answer is that mixed solvents can be used. Neither of the two solvents chosen must cause specific effects upon the rate. Thus a solvent of higher dielectric constant and a solvent of lower dielectric constant, the values of the dielectric constants of the mixtures of which, as a function of composition, have been recorded in the literature, are chosen. Several solvent pairs have been so studied (*6, 7*). Dioxane with a dielectric constant of approximately 2 and water with a dielectric constant of about 78 at ordinary temperatures is a favorite combination. The dioxane-water combination allows a wide variation of dielectric constant, the concentration variation of which over the range from 0 to 100 per cent of dioxane has been studied by Wyman (*7*) at various temperatures. Furthermore dioxane is an inner ether, is inert to many materials, and in addition is soluble in water at all proportions as is indicated by Wyman's dielectric constant studies. Thus, if one wants to decrease the dielectric constant in known amounts one calculates from Wyman's data the percentages of dioxane to put in the mixtures with water at the temperature involved.

Other mixtures for which data occur in the literature (*6*) include CH_3OH-H_2O, C_2H_5OH-H_2O, acetone-H_2O, and sucrose-H_2O.

Suppose it is desired to decrease the rate of reaction, at given temperature and concentration of reactants, between two ions of unlike sign such as NH_4^+ and CNO^- which react to give ammonia. Then an indifferent salt, *e. g.*, $NaNO_3$ could be added in accordance with the extent of lowered rate desired; also if methyl alcohol-water solvent is being used the proportion of water could be increased. The added salt would decrease the effective charge on the reactants thus decreasing the attractive Coulomb force and hence the rate. The increased amount of water would raise the dielectric constant of the medium thus decreasing the attractive Coulombic force and consequently lowering the rate of reaction.

In the following table are summarized the predictions of Coulomb's law concerning the effects upon the rates of reaction between unlike signs of reactants and between like signs of reactants of changing ionic concentration and changing dielectric constant.

	Increase of total ion concentration	*Decrease of total ion concentration*	*Increase of dielectric constant*	*Decrease of dielectric constant*
Effect upon rate of reaction between unlike charge signs of reactants	Decrease	Increase	Decrease	Increase
Effect upon rate of reaction between like charge signs of reactants	Increase	Decrease	Increase	Decrease

DEMONSTRATION

In giving a lecture demonstration, reactions that proceed fast enough to undergo perceptible change during the average lecture time of 50 minutes should be chosen. Reactions of greater rates are very desirable but slower ones cannot well be used. Further, it is best that the reactions used should be such that the rates of change are visually observable.

It is not necessary that quantitative data be obtained in experiments designed to show how reaction rates can be increased or decreased by changing certain variables. Indeed in the short time of a lecture demonstration it would be difficult to obtain precise and accurate data even on reaction rates most amenable to the time allotted and to precision methods of measurement.

In this lecture demonstration what is sought is a dramatic, easily observable, quick, qualitative demonstration of the points in question.

For the above reasons two reactions are used to illustrate the points brought out in the theoretical discussion. The effect of dielectric constant of the

medium can be demonstrated in the following way, using the reaction between the negative bivalent tetrabromophenolsulfonphthalein ion and the negative hydroxyl ion to give the colorless carbinol (*5*). The reaction is thus between ions of like charge sign and the rate should be slowed down by decreasing the dielectric constant of the solvent. At a given temperature the dielectric constants of water-ethyl alcohol mixtures are lower than that of water depending on the proportion of alcohol. We shall therefore choose such a concentration of alcohol as to change the dielectric constant and hence the rate of the fading reaction appreciably. Such concentrations of reactants will be used that the difference in rates in pure water and in water-alcohol solvents are readily observable within the 50-minute lecture period.

Prepare two solutions simultaneously. Solution 1 is prepared by pipetting into a 100-ml. volumetric flask 10 ml. of 10^{-4} *m*-tetrabromophenolsulfonphthalein, 50 ml. of 3 *N* sodium hydroxide, and 25 ml. of ethyl alcohol, and diluting to the mark.

Solution 2 is prepared by pipetting into a 100-ml. volumetric flask 10 ml. of 10^{-4} *m*-tetrabromophenolsulfonphthalein and 50 ml. of 3 *N* sodium hydroxide, and diluting to the mark.

Both solutions are mixed well by inverting the flasks several times and the flasks are then set on the lecture desk for observation. It will be observed that within a period of 30 minutes or so the dye in the pure water (dielectric constant 78.5 at 25°C.) will be almost completely faded. The dye in water-ethyl alcohol mixture (dielectric constant about 67 at 25°C.) will not have faded appreciably but, on standing 12 to 15 hours, will be decolorized. By reducing the proportion of alcohol in the mixed solvent the difference in fading rate in water and water-alcohol solvents can be reduced.

The above reaction cannot be used to demonstrate the salt effect satisfactorily because at the high concentration of sodium hydroxide used for short fading time the ionic strength effect has practically reached its limiting value and further addition of salt does not have a marked effect. A reaction which is amenable to the demonstration of the salt effect is the iodine clock reaction (*8*). The slow rate-controlling step in this reaction is the oxidation of the sulfite ion by iodate ion to the sulfate ion. The iodate ion is reduced to iodide ion which, when all the sulfite ion is used up, is oxidized by excess iodate ion to yield iodine. The iodine with starch gives starch-iodide blue. Therefore, as soon as all of the sulfite ion is used up, the solution which contains starch instantly turns blue. The controlling step of the reaction is again between ions of like charge sign and the rate should increase with increased ionic strength.

Prepare two solutions. Solution 1 contains 2.0 g. of potassium iodate per liter of water solution. Solution 2 contains 0.40 g. of sodium bisulfite and 5.0 ml. of 1.0 *M* sulfuric acid per liter of water solution. Prepare also a saturated solution of sodium chloride.

Into a beaker of convenient size measure 20 ml. of water. Into one 10-ml. graduate measure 10 ml. of solution 1 and into another like graduate measure 10 ml. of solution 2. Invert simultaneously the two 10-ml. graduates containing the two solutions over the beaker containing the 20 ml. of water and note the time (or start a stop watch). Allow the graduates to drain for five seconds and mix the contents of the beaker well by giving the beaker a swirling motion with the hand. In about 140 seconds the solution will instantly become blue in color. Time the appearance of the blue color (stop the stop watch).

Now measure into a clean beaker 10 ml. of water and 10 ml. of saturated sodium chloride solution. Again add simultaneously 10 ml. each of solutions 1 and 2 from graduates and time as before. The blue color will now appear in about 58–60 seconds.

If the 20 ml. of water is replaced entirely by 20 ml. of saturated salt solution and the rest of the experiment is carried out identically as above the time of appearance of the blue color will be reduced to about 20 seconds.

The iodine clock reaction is not well adapted to the study of dielectric constant effects in mixed solvents because all common solvents other than water are chemically active with the reagents of the reaction. Thus dioxane and acetone react with and decolorize starch-iodide blue so that there is no blue color formed when either of these solvents constitutes one component of the mixed solvent. Ethyl alcohol and methyl alcohol affect the starch. For example, when the mixed solvent is 50 per cent by volume ethyl or methyl alcohol the starch visibly forms larger aggregates and in ethyl alcohol is precipitated in string-like fluffs.

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Merry Christmas

THE USE OF PUNCHED CARDS IN THE TEACHING OF QUALITATIVE ORGANIC ANALYSIS

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THE instructor conducting a course in qualitative organic analysis soon finds he has acquired a large number of compounds, each with different chemical and physical properties, and also differing in ease of handling from the student viewpoint. The punched card shown in the figure has been designed to facilitate the cataloguing of such compounds and has proved very valuable in the teaching of such a course.

The card is divided into two types of coding:[1] *direct* for functional groups, solubility, and a "hard-medium-easy" rating for student use, and *numerical* for empirical formulas, physical data, etc. Thus, it is possible to sort the cards rapidly and select for a student a compound with certain functional groups, a boiling point within a given range, and a certain type of solubility. As the student progresses in the course, he can be given more difficult unknowns. Mixtures can be made up easily, for example, by sorting for a given boiling range, and then dividing the selected cards according to solubility groups.

These cards are used primarily for listing the unknowns used in a course. Results of student experiments are typed directly on the card in a condensed form, and these data are then used to evaluate the unknowns as hard, medium, or easy. It has been found advisable to make up mixtures only from the easy compounds.

Reagents may also be listed on the cards, and relevant data concerning the application of these compounds recorded. The cards have also been used for abstracting the literature and outlining lectures. The flexibility of the system—*i.e.*, ease of filing under various headings—makes the cards ideal in this regard. Approximately 10 to 20 literature cards are made up from each issue of *Chemical Abstracts* throughout the year and this record is used for the preparation of lectures. The patent part of Section 10 is especially valuable for commercial separation of mixtures of organic compounds.

Functional Groups (direct coding). These, together with ring structures, elements other than C and H, and a few other direct codings are listed on the upper edge of the card. In most cases separate codings are given to the inner and outer holes. Both shallow and inner punches are used, and a deep punch, of course, for both codings. In a few cases, as for alcohols, ketones, acids, etc., only one coding is given; a shallow punch signifies a monofunctional group, whereas a deep punch is used for a polyfunctional compound (ethanol and ethylene glycol, for example). No inner punch is used in this case.[2] In the case of an ester, such as ethyl benzoate, a deep aryl punch denotes the acid portion as aromatic, whereas the alkyl shallow punch is used for the alcohol portion. With phenyl acetate, a deep alkyl and a shallow aryl punch are used.

From the various codings given, it is possible to sort for most of the ordinary organic compounds encountered in a teaching course. As an expansion feature, six numbers are provided on inner holes, and two more numbers (seven and eight) on the upper right side are placed for future codings.

Solubility Groups (direct coding). These are given on the upper left side and are taken from Shriner and Fuson's classification scheme.[3]

Student Ratings (direct coding). This coding is given on the left side also. When a compound is first assigned as an unknown, the *H* (*hard*) hole is punched. Then, as more students work with the substance and its ease of handling as an unknown becomes apparent, the *M* (*medium*) and *E* (*easy*) holes are punched. Finally, the unknown's rating moves to the *mixture* class. The sorting of a series of cards in this rating should be carried from right to left so that the last (or easiest) rating is dropped first.

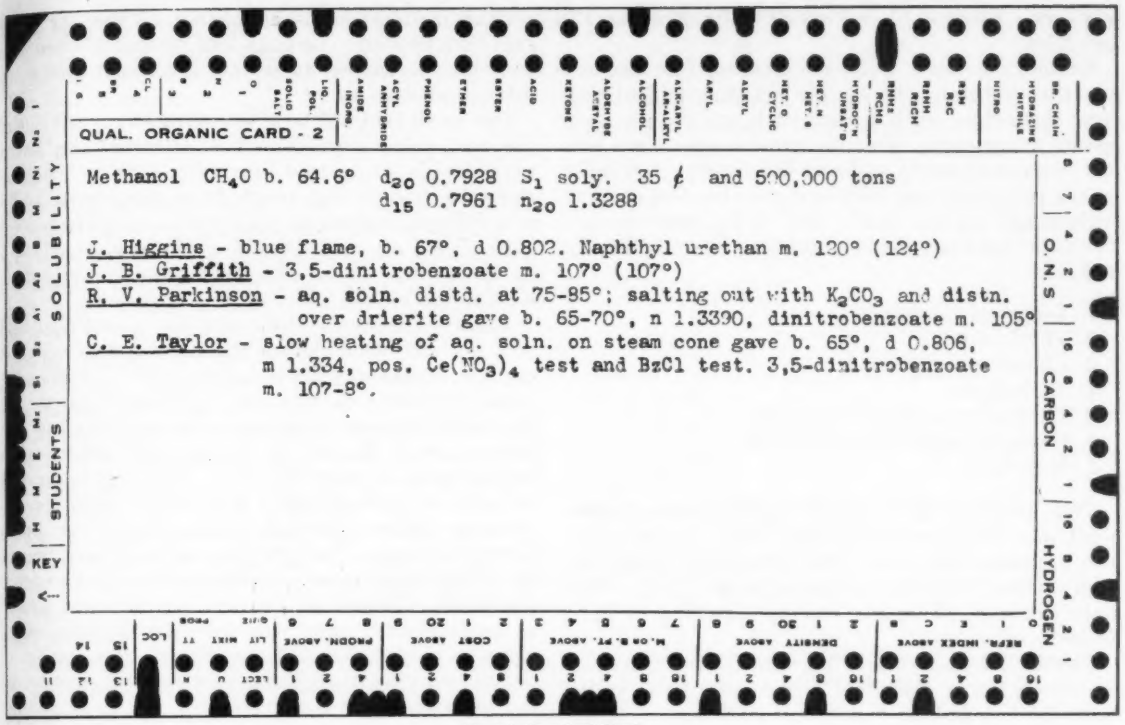
Empirical Formulas (numerical or field coding). This is given on the right side of the card, the number of carbon and hydrogen atoms being coded up to 31.[4] Limited provision is also made for coding the number of the next most frequent element, in the order: O, N, S, Cl, Br, and I. Methanol is coded CH_4O, but acetamide (C_2H_5ON) is added as C_2H_5O only, the presence of N being shown in the direct coding on the top of the card.

[1] The reader is referred to COX, G. J., C. F. BAILEY, AND R. S. CASEY, "Punch cards for chemical bibliographies," *Ind. Eng. News*, **23**, 1623–6 (1945), for a detailed description of coding and sorting procedures.

[2] It will be noted that only one coding (shallow punch) for a *branch chain* is given in the upper right corner. This is done to avoid a deep punch which, combined with a punch in the adjoining side hole, gives a very weak corner which may crease easily. The same precaution is taken on the diagonally opposite corner.

[3] SHRINER, R. L., AND R. C. FUSON, "Identification of Organic Compounds," John Wiley & Sons, Inc., **1948**, p. 59.

[4] A binomial numerical coding is used, rather than the familiar 7-4-2-1 decimal coding; the binomial system allows a greater number of entries to be made in a given number of holes, in contrast to the decimal system. Thus a 7-4-2-1 system is used for 0 to 9 (10 variables), whereas the 8-4-2-1 system ranges from 0 to 15 (16 variables). A 16-8-4-2-1 field has 31 variables. This binomial system is used for all the numerical codings on the card.


QUAL. ORGANIC CARD - 2
Methanol CH4O b. 64.6° d20 0.7928 S1 soly. 35 ℓ and 500,000 tons
d15 0.7961 n20 1.3288
J. Higgins - blue flame, b. 67°, d 0.802. Naphthyl urethan m. 130° (124°)
J. B. Griffith - 3,5-dinitrobenzoate m. 107° (107°)
R. V. Parkinson - aq. soln. distd. at 75-85°; salting out with K2CO3 and distn. over drierite gave b. 65-70°, n 1.3390, dinitrobenzoate m. 105°
C. E. Taylor - slow heating of aq. soln. on steam cone gave b. 65°, d 0.806, m 1.334, pos. Ce(NO3)4 test and BzCl test. 3,5-dinitrobenzoate m. 107-8°.
SOLUBILITY
STUDENTS
KEY
O. N. S
CARBON
HYDROGEN

Sample Punched Card

In an earlier set of punch cards a numerical coding was made for the molecular weight but the empirical formula is more desirable.

Physical Data. These are given in numerically coded fields on the lower edge of the card. They are set up in the outer holes only.[5]

The *refractive index* field is set up in 31 steps of 0.01 each, ranging from 1.31 to 1.61. In the *density* field, the first 25 intervals of 0.02 each for the range from 0.60 to 1.10, and the last six intervals of 0.10 range from 1.20 to 1.70 are used. The *melting* and *boiling* points (the latter are at 760 mm.) are given in 31 steps ranging from 0° to 310° in 10° intervals. The choice between a boiling and melting point is given by the listing of a compound as a liquid or solid, respectively, on the upper edge of the card.

Miscellaneous Fields. These are given also in the lower portion of the card. In addition, a series of numbers, from 11 to 32, is given as an expansion region for future direct codings. The *cost* field is set up exponentially, the cost being given in 2^n cents per 100 grams, where n ranges from 1 to 15. This type of coding gives a wide variation in values. The standard price used is that given in the Eastman Organic Chemicals Catalogue for a c.p. compound. *Production* is also listed exponentially, the yearly production being given in 10^n tons, where n ranges from 1 to 7. This field gives the instructor an idea of the commercial use of the simple laboratory chemicals. *Location* is used to denote whether a chemical is on the shelf in the instructor's laboratory or in the stockroom (deep and shallow punches, respectively).

Miscellaneous Direct Coding. A series of three holes to the right of the production field serves as main codings for the use of the cards. The shallow *U* (unknown) punch is used to list all single compounds used as student unknowns; on these cards are listed the students' data, reactions, derivatives, etc. The inner *Mixt* (mixture) punch is used for student data concerning the separation of mixtures. Once a mixture is separated into the several components, the data for the latter are then listed on "unknown" cards. Several functional groups or solubility groups may be coded on a "mixture" card, as phenol and acid, or A_1 and A_2 solubility groups. *Reagents* (*R*) are listed on cards in a manner similar to unknowns but the data recorded are relevant only to the action of the reagent. Chloroacetic acid, for example, would be coded as a reagent, and also under phenols, with which it forms derivatives. It would not be coded, in its consideration as a reagent, as an alkyl acid. However, it may be listed on a separate card as an unknown and, in this case, the normal functional groups would be listed. *Test tube experiments* (*TT*, a deep punch) are a series of preliminary experiments given to the students to familiarize them with functional groups or separation of

[5] It is easy to sort cards in sequence when the outer holes are used, but very difficult to do such sorting for the inner holes. Therefore, all important numerical fields are coded in the outer holes only.

mixtures. These cards are coded *TT* and *U*, or *TT* and *Mixt*.

Lecture, *Literature*, *Quiz*, and *Problem* (the last two are inner punches, whereas *Lit* is a deep punch) concern themselves with general subjects rather than specific compounds. For this reason a specialized subject index is set up by utilizing the density, melting or boiling point, and cost fields as a main heading and two subheadings (0–31, 0–31, and 0–15 respectively). The main headings, listed under the "altered" density field, are:

1. Laboratory technique
2. Physical data
3. Classification reactions
4. Derivatives
5. Physical separation of mixtures
6. Chemical separation of mixtures
7. Miscellaneous

Thus, paper partition chromatography is listed under 5-4-0, that with paper strips under 5-4-4, that on cellulose columns 5-4-5, etc. This numerical coding has been confined thus far to a maximum of 7-15-15. The main headings, of course, can be expanded to 31 if desired.

Similarly, the carbon and hydrogen fields are used alternatively for a *Date* field, the decades being coded under Carbon and the year under Hydrogen.

Although these two special numerical fields are set up for literature or lecture cards, the other codings can still be used in the usual manner. Thus, for paper partition headings, sortings can be made when this technique is applied to certain functional groups, as acids or phenols, etc.

One small field will be noted on the lower left side of the card, titled Key. This field of three holes (seven variables) is set up to distinguish one set of cards, as those described in this article, from other types, coded in a different manner for other purposes. The present type of card is punched at the center hole, as indicated by the arrow. Thus, with this master key, seven sets of cards can be used with no fear of confusion.

Finally, the reader will note an *alphabetical* field (*O I E C B*)[6] below the refractive index field. This is used for the initial letter of a compound. In an earlier card, the author coded a series of three fields for the first three letters of a given compound, but this was seldom used. Hence, the present field has been restricted to the initial letter only. It is very interesting to note how much simpler it is to sort for the functional group or empirical formula rather than for an alphabetical heading. The latter may be rather ambiguous, as in the listing of amyl alcohol under A or under P (pentanol). Esters can be sorted functionally, whereas alphabetically they would be listed under ethyl, methyl, phenyl, etc. A functional cataloguing of organic compounds in handbooks or catalogues would in some ways be more desirable than the present alphabetical type.

[6] Cox, G. J., C. F. Bailey, and R. S. Casey, *op. cit.*

A TEMPERATURE-EQUILIBRIUM DEMONSTRATION

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We have felt an acute need for good demonstrations in the teaching of equilibrium principles to freshmen, since the freshmen seem to have a hard time grasping the idea from the study of abstract equations.

This demonstration makes use of the colored cobaltous complexes in a mixed solvent to show the dependence of some equilibria on temperature. The solution is red at room temperature, and turns blue upon warming, reverting to red again upon cooling.

The reaction may probably be represented by the equation:

$$\underset{\text{blue}}{CoCl_4^{--}} + 6\ H_2O \overset{\text{alcohol}}{\rightleftharpoons} \underset{\text{red}}{Co(H_2O)_6^{++}} + 4\ Cl^- + \text{heat}$$

The exact structures of these complexes are none too certain. It has been reported[1] that the blue complex has the general formula CoX_4^{--} in solutions with excess halide, but a blue complex CoX_2 has also been reported in solutions without excess halide.[2] The red complex is usually considered to be the normal hydrated cobaltous ion.[2]

The demonstration may be performed as follows. Dissolve 0.2 g. of $CoCl_2 \cdot 6H_2O$ in 15 ml. of 95 per cent ethyl alcohol. This concentration is not critical—use enough to get a pleasing color. Add water, slowly, until the blue solution turns red. This will take 1 or 2 ml., depending on the exact amount of $CoCl_2 \cdot 6H_2O$ used and the purity of the alcohol. Heat the solution to make sure it works. If the transition temperature is too high, a little alcohol will lower it; water will raise it. Seal the solution into two ampoules to prevent evaporation, and heat one to show the color change.

[1] Barvinok, M. S., *J. Phys. Chem.* (*U.S.S.R.*), **22**, 1100–7 (1948); **19**, 612–16, 793–7, 1028–33 (1949).

[2] Bobtelsky, M., and K. S. Spiegler, *J. Chem. Soc.*, 1949, 143–8.

A QUANTITATIVE SODIUM FLAME TEST

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A SIMPLE spectrophotometer was improvised as follows. A square of polaroid was fastened in front of the telescope objective of a Bunsen-type spectroscope. A wedge of cork between the lens mounting and the prism table sufficed to hold it in place. A small disc of polaroid was cut to fit the eyepiece tube and was held in position in the lens mounting.

An ordinary brass protractor was soldered to a split tube of such size as to fit snugly on the focussing tube of the telescope. A narrow sheet of sheet metal bent about the eyepiece tube, with one long arm extending out over the protractor scale to serve as a pointer, completed the instrument (Figure 1).

The method of producing the flame was a modification of the qualitative procedure devised by A. Ehringhaus,[1] in which a roll of filter paper was substituted for platinum wire as a carrier for solutions of flame-coloring substances.

Small squares of filter paper, 3 × 3 cm., were used in these tests. Glass tube holders made of 15-cm. lengths of 4-mm. (internal diameter) tubing were bent to an obtuse angle of about 30 degrees at 5 cm. from one end. A square of filter paper was rolled about a small nail and inserted half way into the short arm of the bent tube. The nail was removed and a 4-cm. length of No. 2 twisted iron picture wire inserted in its place. The wire was bent so as not to slide out of place in the roll, and allowed to project about 3 mm. beyond the paper. This wire heats in the flame and

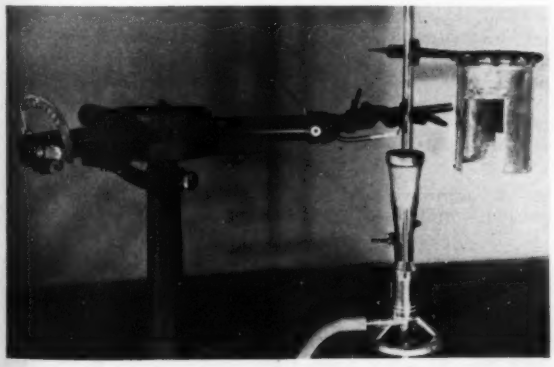

Figure 1

accelerates the vaporization of the solution. A Fisher high-temperature burner was used and was protected from drafts by a sheet metal shield. In all of these tests the same height of flame was found necessary.

The tube thus prepared was filled half-full with the test solution and placed in a holder made of a stand and an adjustable buret clamp holding a slotted rubber stopper of such size as to firmly grip the long arm of the solution tube. The spectroscope slit was placed 15 cm. from the flame. A wide slit covering three divisions of the Bunsen scale was used in these experiments.

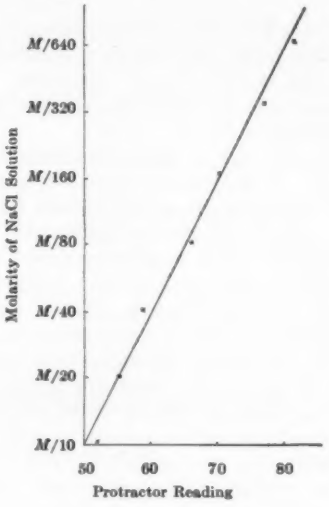

Figure 2

The calibration curve in Figure 2 was based on the extinction angles of flames produced by solutions of sodium chloride of known molar strength. An approximately linear relationship was established as shown. These results were obtained in a laboratory with ordinary lighting.

Using this method a sample of c.p. potassium iodide gave a sodium content of 0.17 ± 0.03 per cent. The manufacturer's marking was "Na less than 0.03 %."

An interesting test with magnesium sulfate, manufacturer's mark, "0.05 % Na," showed 0.02 per cent; but when acidified with Na-free HCl, it tested 0.84 per cent.

Precautions. The extinction readings must be made rapidly, because the flame intensity increases with the gradual increase in solution concentration due to evaporation. The appearance of crystals on the paper makes the readings useless.

The wick should be inserted at the same height from the burner top.

One-tenth molar sodium chloride solution was the strongest that could be used. Stronger solutions should be proportionately diluted. Best results come in the range indicated by the graph.

[1] *Centr. Mineral. Geol.*, 1919, 192.

A LITERATURE SEARCH ASSIGNMENT[1]

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No one who has done any significant research in chemistry will doubt the need for a good search of the literature. Various problems associated with such a search have been discussed from time to time in This Journal and elsewhere.[2, 3]

Many schools offer a course in chemical literature although some do not require it of their undergraduate majors. Many other departments feel that a formal course in chemical literature is undesirable for budgetary reasons or because it would take time which cannot be spared from a crowded program. It has been stated with considerable justification that a course about chemical literature is like a course about research[3] and is of little interest unless correlated with live problems.

Professor Sillén has discussed this problem in public lectures ("The Chemist's Brain—A Housing Problem") which unfortunately has only been published in Swedish.[4] He points out that while the fund of knowledge is increasing exponentially with time, the material which a student remembers decreases exponentially with the time elapsed since it was studied. As a result, no chemist can possibly know more than a minute fraction of the available chemical knowledge. However, it is still possible for him to know how to find a substantial fraction of the total knowledge although some phases will remain unintelligible for lack of suitable training. Sillén makes a plea for more attention to learning principles and methods of finding information and less attention to memorizing unrelated facts. He describes a method of literature training in use at Stockholm's Technical University. The students are set small literature problems from their first year: "What is known about the formula for chloride of lime?"; "Outline safety precautions for the use of perchloric acid"; "How are the atoms arranged in red lead?" which require reference to information beyond that found in the ordinary textbooks. This is followed by problems in synthetic inorganic and organic chemistry which are not presented in a laboratory manual but must be looked up in the original. In this way, the habit of referring to the larger handbooks and abstract journals for needed information is instilled along with the basic principles of chemistry.

One common approach to this problem, which has been used by the author, is to assign term papers in graduate and "fifth" courses. These assignments require that the student refer to the original literature although they sometimes turn out to be based upon a single review article. Since, for many of the students, such a term paper is their first encounter with the original literature, it seems desirable to give some introduction to the library and the bibliographic aids which are available. Such an introduction can be made in a single lecture followed by a tour of the library facilities.

A follow-up of this introduction to the library is logically a specific assignment involving the finding of some original data. Obviously each student must have an individual assignment, both to insure independent work and to reduce the wear and tear on the library facilities. Individual assignments present no problem in small classes but the task of preparing twenty to fifty different problems may prove to be a sufficient additional burden to result in its postponement "until I have more time!"

In order to reduce the labor of making up and grading literature search assignments in a course on the colloid chemistry of high polymers, the following form was prepared and duplicated.

Literature Search

Student's name_____

Locate the following information and outline the chain of references that you followed to obtain it. Do not include blind leads. Indicate any article which you were unable to see and if possible give a reference to an abstract of this article. Report your findings on this page, giving title of article, and journal reference or patent number.

I. The first paper or patent describing the production of_____.

(3-in. space)

II. A recent review article dealing with the properties, production, or use of this material.

(1-in. space)

III. Tabulate, with reference to the original source, at least three physical properties of this material.

A different high polymer was entered on each form and the students drew their assignments at random from the sheaf of forms to avoid charges of favoritism since some assignments are inevitably easier than others.

Other branches of chemistry would require obvious modifications of this wording. One might use nearly the same form for industrial chemicals or pharmaceutical products. A rather different form would be necessary to give training in searching for preparative methods. In analytical chemistry one could ask for a

[1] Presented at the Pacific Northwest Regional Meeting of the American Chemical Society, Seattle, June 8–9, 1951.

[2] For example see: Egloff, G., *et al.*, J. Chem. Educ., **20**, 493, 587 (1943); *ibid.*, **21**, 615 (1944). Also, "Searching the Chemical Literature," Staff of Industrial and Engineering Chemistry, A. C. S., 1951.

[3] Meldrum, W. B., and T. O. Jones, J. Chem. Educ., **20**, 592 (1943).

[4] Sillén, L. G., *Elementa*, **32**, 161 (1949).

review of the methods for determining a given compound or element, a reference to the original paper describing one of these methods, and a presentation of the range and limits of precision of a modern version of the same method. In physical chemistry one could similarly ask the students to locate references to methods for determining a physical property.

This form is similar to that reported by Emling for a patent assignment.[5] His assignment has been used with favorable results as a sequel to the one presented here since a more detailed knowledge of the type of information contained in patents is desirable for all chemists who are going into industry, or who expect to teach chemists who will go into industry.

The literature search assignment described in this paper may be of questionable value if it is not followed up by further problems such as arise in the preparation of term papers and ultimately in the preparation of research theses. Its main function is to help the student to get started so that he may be expected to do a better job on his first term paper.

[5] EMLING, B. L., J. CHEM. EDUC., 28, 133 (1951).

A COMPARISON OF CRITERIA FOR THE REJECTION OF MEASUREMENTS

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THE rejection of measurements is dealt with in almost all elementary texts in quantitative analysis, but the treatments leave much to be desired. Some presentations of this subject appear contradictory to others, some are inapplicable to most chemical data, and some are even incorrect. It is the purpose of this paper to discuss and compare some of the accepted criteria for rejection of data, and to sum up the principles on which such rejection may properly be made.

In a set of measurements on a single quantity, if one measurement is "discordant" with others of the set, the question arises as to whether the discordant measurement should be discarded or retained.

If it can be decided that some specific error may have been made in obtaining the doubtful observation, then it should be rejected, regardless of how it stands in relation to the other observations of the set. Certainly, no measurement should be retained which is experimentally questionable in any respect. This is probably the safest and most reliable basis for rejection of data; further, it seems to be the only generally applicable one.

Using the concept of a "statistically significant difference" (*6*) many authors state that it is possible to reject a discordant measurement for no other reason than that it differs considerably from other measurements of the set, even though the discordant one may be experimentally no more questionable than the others. The justification for such rejection is that a large difference between any one observation and others of the same set would not be expected to occur very frequently, and that if such a large difference exists, it is probably due to some (unknown) error made in obtaining the questionable measurement.

In attempting to apply this statistical principle to discarding measurements, it is necessary to decide how large the difference must be before rejection may be made, and it is in fixing this minimum difference that difficulty arises, for the experimenter is faced with two contradictory objectives:

(1) If the investigator wishes to be quite sure that the difference between the questionable observation and the others of the set is statistically significant, then the minimum difference required for rejection must be so large that many measurements which are quite discordant cannot be excluded because the differences are not great enough to meet the criterion for rejection. In other words, setting the minimum difference high enough to assure good statistical significance causes erroneous measurements to be retained too high a proportion of the time. (This is called an "error of the second kind" by statisticians.) This is very bad, for discordant observations have a great effect on the mean and may cause considerable error if retained.

(2) On the other hand, if the minimum difference for

rejection is made too small, then valid data may be rejected too high a proportion of the time. (This is called an "error of the first kind" by statisticians.) The wastefulness of this practice is not its worst evil. The error of the mean is actually increased in thus reducing the number of observations, and the increase is appreciable when the number of observations is small. Further, such rejection may lead to retention only of values that are spuriously close together, and therefore to a claim for spuriously high precision, which may be extremely misleading.

Various criteria for rejection of measurements differ principally in the relative extent to which each of these two opposing objectives is emphasized.

In all the following procedures for rejection, the distribution of the observations in the set (called the "sample") is examined. If this distribution corresponds reasonably to that expected in a good random sample drawn from the same normal population of measurements as the actual sample, then all measurements in the actual sample are retained. If, on the other hand, the actual sample shows an improbable distribution, the outlying observation is rejected. This approach is valid in principle, but when applied to measurements of the chemist, which are almost invariably few in number (*i. e.*, two to six), the conclusions are very uncertain. The reason for this is that the inference as to the nature of the normal population from which the actual sample was drawn becomes more uncertain as the number of determinations decreases.

The following criteria for rejection of measurements are examined in detail and evaluated in the light of the above considerations.

The "2.5d" Rule. Several authors (*3*, *7*, *9*) use the following principle for rejection of a discordant measurement. This rule seems to be gaining in popularity, even though it is incorrect. There must be at least four measurements in the sample, but not more than six or eight. Compute the mean (a_{n-1}) and the average deviation (d_{n-1}) of the observations in the sample, omitting the questionable value. If the deviation (x) of the questionable value from the mean (a_{n-1}) is more than 2.5 fold greater than the average deviation of the closely grouped values (*i. e.*, more than 2.5 d_{n-1}), the discordant observation may be rejected.

In justification, it is stated that if the deviation of an observation exceeds this limit, then the chances are only about 1 : 20 or less that the discordant observation is really a valid member of the population from which the remaining members of the sample are drawn. The chances are 19 : 20 or greater that some definite (unknown) error is involved in the discordant observation, which may therefore be safely rejected.

This justification is based on the statistics of samples of large numbers of determinations, drawn randomly from a normal population, and is extended to small samples without proper compensation therefor. Further, in estimating the average deviation from the most closely grouped observations, a "selection" is made, and the sample is no longer a random one. This criterion is not a good one; the limit set for rejection is much too low and leads too frequently to rejection of valid data and to erroneously high estimates of precision (*i. e.*, to errors of the first kind).

This principle is applied to a few selected cases in Table 1, where the "discordant" values are italicized in each case. In the first three cases rejection of the italicized values seems arbitrary, yet the criterion calls definitely for rejection in every case. The fourth case might be argued to be a "borderline case," yet the rule conclusively rejects the italicized measurement. In the fifth case the italicized value is not even the most discordant, yet according to the 2.5*d* criterion, it is almost a borderline case.

TABLE 1
Application of the 2.5d Rejection Criterion to Specific Samples

Sample	*Measurements*[a]	*Mean of "good" measurements*	*Av. dev. (d_{n-1}) of "good" measurements*	$\frac{x}{d_{n-1}}$[b]
1	1.1, 1.2, 1.2, *1.3*	1.167	0.045	3.0
2	1.1, 1.2, 1.3, *1.4*	1.20	0.067	3.0
3	1.1, 1.1, 1.2, *1.3*	1.133	0.045	3.7
4	1.0, 1.2, 1.2, *1.5*	1.133	0.089	4.1
5	*1.0*, 1.2, 1.2, 1.5	1.30	0.133	2.3

[a] The italicized measurement in each series is considered the discordant one.
[b] x = deviation of italicized measurement from mean of "good" measurements.

The distribution of x/d_{n-1} was considered difficult to find theoretically, but it was found experimentally as follows: Two hundred random samples of n determinations each were drawn from a table of normal random numbers, distributed about a true mean of 2 and having a standard deviation of 1 (*i. e.*, an average deviation of 0.80) (*6*). For each sample, there was computed the average (a_{n-1}) and average deviation (d_{n-1}) of the n-1 close determinations, and the deviation (x) of the discordant observation from a_{n-1}. The ratio x/d_{n-1} was then found for each sample.

A cumulative distribution of the percentage of samples having x/d_{n-1} at or above certain values is shown in the figure for sample sizes having n = 3, 4, and 5. Study of the figure shows that for sample sizes with n = 4, about 86 per cent of the samples have x/d_{n-1} above 2.5, while for n = 5, 82 per cent have x/d_{n-1} above 2.5. These proportions are much greater than the previously mentioned 5 per cent predicted from the statistics of large samples. Following this rule would result in rejecting valid measurements about 5 out of 6 times for samples of 4 or 5 determinations!

Actually, considerably more work was done with these experimental samples than is reported in the figure. The average (a_n) and average deviation (d_n) of all the determinations in each sample were also computed. It was found that a_n of all the determinations in each sample was more often closer to the true mean than a_{n-1} of the closely grouped determinations in each

sample, which is in accord with theory, since the mean of n determinations is more reliable than that of n-1 determinations. Also, it was found that the average deviation (d_{n-1}) of the close determinations was several times less than the average deviation of the population from which the samples were taken; or in other words, that d_{n-1} furnished an erroneously high estimate of the precision.

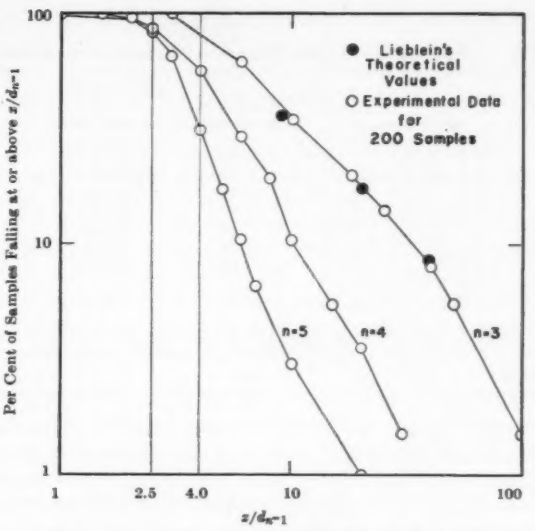
These estimates are rendered less favorable for the argument if it is considered that the sample may actually contain erroneous values a certain proportion of the time. (It must be remembered that the curves in the figure are for samples consisting of equally reliable observations, all drawn from the same normal population.) In any worth-while chemical analysis, however, this proportion of erroneous values must be small, and therefore cannot have much effect on these estimates. If it is argued that this proportion of erroneous results might be great enough to influence the above estimates appreciably, then such analyses are not worth considering at all; nothing can aid in the interpretation of analyses where it must be supposed that gross error occurs frequently. The problem at hand is concerned with the rejection of occasional errors. For these reasons, the above estimates and those in the figure may be considered to be good approximations of those encountered in actual practice.

Tests on the distribution of a_n, a_{n-1}, and d_n indicated that the samples were properly and randomly chosen. As a further check on the correctness of these estimates and conclusions, Youden and Lieblein have studied the problem of two out of three determinations experimentally and theoretically, with essentially the same results as stated above. Their work is summarized very interestingly in a short article entitled: "The fallacy of the best two out of three determinations" (*11*). A few points are shown in the figure for the distribution of x/d_{n-1} for samples with $n = 3$, taken from this article.

The "4d" Rule. Almost all earlier texts in quantitative analysis and some of the newer ones set the limit of the preceding rule at $4d_{n-1}$ instead of $2.5d_{n-1}$. The justification given for the limit of $4d_{n-1}$ is that if a discordant observation in a set differs by $4d_{n-1}$ from the mean of the others, then the chances are very high that some definite error exists in the discordant observation. The probability that the discordant observation is a valid member of the same population as the other measurements of the set (*i. e.*, the probability of making a statistical error of the first kind) is quoted by various authors as being only 1.4 to 7 in 1000 (*9, 12*).

If these probability estimates were correct, it could be argued that the $4d_{n-1}$ limit is so high that statistical errors of the second kind (*i. e.*, retention of erroneous values) would occur too frequently. It is this argument which has led (incorrectly) to the adoption of the $2.5d_{n-1}$ limit in preference to $4d_{n-1}$ by some authors.

Examination of the figure shows that when four observations are made on a quantity, the ratio of the deviation of one from the average of the three close



Distribution of x/d_{n-1} for Samples with n = 3, 4, and 5

ones may exceed four times the average deviation of the three close ones almost 60 per cent of the time. For five observations, the ratio may exceed four about 36 per cent of the time. Use of this rule would result in errors of the first kind and rejection of good measurements around half of the time, for sets of four or five determinations. Rejection cannot be made with any reasonable degree of confidence using this rule, and it should not be employed even roughly for the rejection of one in a small number of determinations.

The Chauvenet Criterion. This test is much used in radiochemistry for the rejection of nonconforming counting measurements (*1*). The procedure for application of this rule is as follows: (1) Find the mean and probable error (0.6745 times the standard deviation[1]) of the whole series of measurements, including the questionable one. (2) If the ratio of the deviation of the discordant observation to the probable error is greater than R, the questionable determination may be rejected. The value of R is dependent upon the number of observations in the set and is given in tables (*2, 10*).

Chauvenet's rule is based upon the statistics of a large number of observations, and is in theory quite versatile, for it may be applied successively to a series of determinations to eliminate more than one discordant observation. Investigation of this criterion, however, shows that it is not much good for any number of determinations.

The rule is devised so that if a discordant measurement falls at the limit for rejection, then it is just as likely to be a valid member of the series as not, and there is appreciable confidence neither in retaining nor

[1] Chauvenet defines his standard deviation as the square root of the sum of the squares of the individual deviations divided by one less than the number of determinations. It is known (*6*) that this gives a biased underestimate of the true standard deviation of the population from which the sample is taken.

TABLE 2
Application of the Chauvenet Criterion to Specific Cases

Case	*Measurements*[a]	*Mean of all measurements*	*Standard dev. (s) of all measurements*	$\frac{x^b}{0.6745s}$	*R, for rejection*
1	1.1, 1.2, *6.1*	2.80	2.86	1.72	2.05
2	1.1, 1.2, 1.2, *3.3*	1.70	1.07	2.22	2.23
3	1.1, 1.1, 1.2, 1.2, *1.3*	1.18	0.084	2.12	
4	1.1, 1.1, 1.2, 1.2, *1.4*	1.20	0.123	2.42	
5	1.1, 1.1, 1.2, 1.2, *1.5*	1.22	0.164	2.56	2.44
6	1.1, 1.1, 1.2, 1.2, *1.7*	1.26	0.251	2.60	
7	1.1, 1.1, 1.2, 1.2, *1.9*	1.30	0.339	2.63	
8	1.1, 1.1, 1.1, 1.1, 1.1 1.2, 1.2, 1.2, 1.2, *1.3*	1.16	0.071	2.93	2.91

[a] The italicized measurement in each series is considered the discordant one.
[b] x = deviation of the italicized measurement from the mean of all measurements.

in rejecting this questionable measurement. This disadvantage exists for a series of any number of determinations. Even for a large number of observations (see case 8, Table 2), rejection of values falling at the limit would seem spurious.

An even more serious error occurs when the rule is extended to a small number of determinations, due to the large effect which the discordant observation may have on the estimated mean and probable error. In fact, the R limits given for rejection of one measurement in a group of three or four is never exceeded in practice, no matter how discordant the measurement. The following example may make this clear for the case of four determinations.

Suppose four determinations on a quantity give y, y, y, and $y + 4q$, where the last is assumed discordant. The mean is then $y + q$, the probable error is $1.3490q$, and the deviation of the discordant observation is $3q$. Hence, the ratio of the deviation of the discordant observation to the probable error is $3q/1.3490q = 2.224$, no matter what the value of q. The value of this ratio for any other set of measurements, where the first three are not identical, cannot be greater than 2.224. The limit, R, given for rejection of one out of four determinations, is 2.23 (see Table 2), greater than the maximum attainable. For three determinations, the difficulty is much worse; the limit for rejection is 2.05, whereas considerations similar to those above show the maximum attainable value is only 1.71. The specific cases 1 and 2 in Table 2 further illustrate the failure when the rule is applied to three or four determinations.

Even for five determinations, the computed ratio may exceed only slightly the value of R set for rejection, and computations must be made quite precisely, for rejection or retention of a questionable value is based on rather small differences in the ratio. This is illustrated in Table 2 by application of the rule to the selected cases 3–7, inclusive.

For these reasons, the Chauvenet criterion should not be used as a basis for rejection of measurements.

The Q Test. In an excellent and recent paper (*4*) on simplified statistics for small numbers of observations, Dean and Dixon have described a very simple test, called the Q test, which is performed as follows: (1) Arrange the determinations in ascending order of magnitude. (2) Calculate the difference between the discordant measurement and its nearest neighbor, and divide this difference by the range (*i. e.*, the difference between the largest and smallest measurement), calling the quotient Q. (3) If Q exceeds certain tabulated values (*4*) which depend upon the number of determinations, the questionable measurement may be rejected with 90 per cent confidence that some definite error is involved in this measurement which was not involved in the other measurements of the set. This rule and its confidence limit are statistically correct. Further, it possesses the advantage of simplicity over other good rules for the rejection of outlying observations (*5*, *8*), even though it may be less efficient in some cases.

However, application of this criterion to sets of small numbers of observations (*i. e.*, three to five) does not allow rejection of an outlying result unless the discrepancy is quite large. A divergent observation would be intuitively suspected considerably before this test would allow its rejection. As stated by Dean and Dixon (*4*), these rules are good primarily as justification for rejection of grossly erroneous values. Because of the large difference required for rejection, errors of the second kind occur, and erroneous values are retained too high a proportion of the time. This is a severe defect if the arithmetic mean of the set of observations is computed, for the mean is greatly influenced by retention of a discordant value. Dean and Dixon have suggested use of the median instead of the arithmetic mean to avoid the effect of extremal values when the latter are suspected but cannot be rejected with confidence. There is a disadvantage in using the median instead of the arithmetic mean, since the median is a less efficient measure of the true central value of the population from which the sample of observations is taken. This was apparent from the data summarized in the figure. For samples containing 3, 4, and 5 determinations, the median was found experimentally to be farther from the true mean more often than the arithmetic mean of all determinations in the samples. This, however, is simply a part of the price which must be paid to reduce the effects of a possibly erroneous observation.

By setting the confidence limits lower than 90 per cent (*i. e.*, to 75 per cent, or even lower), the possibility of making errors of the second kind and retaining erroneous values would be decreased. However, it is recognized by statisticians that such low confidence limits have little meaning when applied to a single sample of a small number of observations. The result of a chemical analysis is usually important, and the recipient of this result usually wants better assurance than three chances in four or so that the result is as stated, and not something considerably different.

Conclusion. Of the various commonly used criteria for rejection of outlying observations examined in this paper, some are statistically incorrect when applied to small samples of only three to five determinations, which is the number usually dealt with by analytical chemists. The "2.5*d*" or "4*d*" rules have limits for rejection that are far too low and lead too frequently to rejection of good data with a loss in accuracy, and to spuriously high estimates of precision. Chauvenet's criterion does not allow rejection of any result in samples of three or four determinations, no matter how discordant, and is of questionable use even in samples of five determinations.

The recently proposed Q test is perhaps the simplest of correct statistical procedures for the rejection of extremal observations, but even this is not very useful when applied to samples of a small number of observations. In order to be fairly certain that the rejected value is really in error, the limits for rejection must be so great that erroneous values are retained too high a proportion of the time. Samples of only three to five determinations are so small that only the roughest guesses of the distribution of the population may be made, and for this reason, correct statistical rules are useful only to justify the rejection of grossly discordant observations.

If the standard deviation is known for the population from which the sample is drawn, the performance of these rules is better and the limits for rejection of outlying values are smaller (*4*). Thus, the experienced analyst, who knows within what range his determinations should fall on the basis of many similar analyses performed by himself or others in the past, may use these tests to reduce the effect of unknown chance errors which arise infrequently. In other words, the experienced analyst bases his judgment upon a disagreement between his known expected precision and that which he has achieved, rather than upon a theoretical expected precision poorly estimated from the small and possibly faulty sample at hand. The student or beginner does not have this experience and does not know the precision of which he is capable. These rules should be applied with great caution and reservation by all except the experienced worker.

These criteria are attempts to arrive at a decision to retain or reject data without employing the knowledge or judgment of the investigator, and as such, they seem attractive, especially to the novice, but they must be unsatisfactory. The only safe and good basis for elimination of a discordant observation is knowing or finding that some definite error has influenced this observation; any other basis is open to question. If one measurement in a small number of measurements is discordant, it should not be arbitrarily discarded, for this has the same effect as arbitrarily bringing it into conformity with the others. Instead, the reason for the discrepancy should be found, or enough additional determinations should be performed to give good assurance that the questionable observation is really wrong.

The following procedure is suggested for interpretation of a sample containing three to five observations, if the precision is considerably lower than expected, and if one observation is considerably different from the others of the set. (1) The data leading to the discordant observation should be retraced to see if some definite error might have been made in obtaining this observation. If such an error is found, the observation may be rejected. (2) If no definite error may be associated with the questionable determination, the Q test should be applied. If this test calls for rejection, such rejection may be made with 90 per cent confidence that an undiscovered error exists in the outlying observation. Performance of additional analyses, if possible, increases the efficiency of this test. (3) If the Q test does not allow rejection of the extreme value, the median of the observations should be used, rather than the arithmetic mean.

ACKNOWLEDGMENT

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A MACRO GAS BURET

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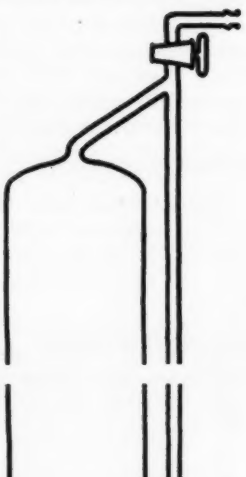
LITHIUM aluminum hydride is showing itself to be a remarkable reducing agent by the ease with which it attacks many linkages and by the very high yields which are frequently obtained. Since this new hydride is easily handled in ether solution and has been shown to react safely with many functional groups,[1] it lends itself well to undergraduate senior research.

One of the senior problems undertaken at Illinois Institute of Technology was to determine the optimum conditions for the reduction of chloroacetic acid to ethylene chlorohydrin. In this study gasometric data were desired to confirm and supplement the results of other types of measurements. This paper describes the construction and calibration of the macro gas buret which was used in this problem and which has since proved a generally useful piece of laboratory apparatus.



The high vapor pressure of ethyl ether (usually the reaction medium of choice) and the desirability of occasionally performing further operations on the products made it virtually impossible to use the conventional "Grignard machine." It was found possible to overcome these difficulties, however, by increasing the scale of operation, using a dry-ice trap to remove ether vapor from the hydrogen, and collecting the hydrogen by displacement of water.

A large carboy, such as that mentioned by Arnold and co-workers,[2] was first used. This was found to be unwieldly for continuous use and was discarded in favor of a graduated, 150-cm., 100-mm. diameter glass tube. The present design is a refinement of the latter form and offers greater convenience together with higher accuracy.

The figure shows the arrangement of the buret with its side tube by which the height of the water in the buret is measured. The body of the buret is made of 100-mm. Pyrex tubing. The side tube, of 8-mm. Pyrex tubing, is attached to the center of the top of the buret by one leg of a Y-tube and to the bottom by a solid stand-off tube 15 mm. long. The height (*i. e.*, the capacity) is a matter of preference. The authors have found a height of about 150 cm., giving a capacity of approximately 10 liters, very satisfactory. None of the dimensions is critical; it is only important that the buret can be filled by vacuum (water aspirator) with no air spaces remaining up to the stopcock.

A length of steel measuring tape, not shown in the figure, is fastened against the side tube. Calibration is easily carried out by filling the apparatus with water and then reading the steel rule after successive volumes of water have been withdrawn. The best way to control this flow of water out of the buret is by means of a stopcock fitted to the bottom of the buret through a No. 15 rubber stopper. During this operation, the side tube must be connected to the bottom of the buret by a short piece of rubber tubing and a short piece of glass tubing inserted through a second hole in the rubber stopper.

Weighing the water withdrawn would give the most accurate results for the calibration. However, such great accuracy would be entirely unnecessary for most purposes. Careful measurement of the water with a calibrated 100-ml. or 200-ml. volumetric flask was found to be convenient and of sufficient accuracy. From the calibration data a table of height versus volume was constructed.

The accuracy of the measured volume is limited by the error made in reading the meniscus. It happens that the scale behind the side tube is conveniently magnified by the water so that the meniscus can easily be read to the nearest mm. This means that the volume can easily be known to the nearest 8 ml. Experiments have shown that interpolation between the calibration points produces less error than reading the meniscus to the nearest mm. If 5 liters of gas are collected (about half the capacity of the buret), the error is less than two-tenths of one per cent. It is possible, of course, to obtain greater accuracy.

In use, the buret stands in a 4-liter beaker and is supported by three three-hole rubber stoppers each of which has glass tubing twice the length of the stopper in every hole. These pieces of glass tubing act as guides down through which the bottom of the buret fits. The top of the buret is clamped to a tall ring stand. The 4-liter beaker has an overflow which keeps the water level approximately 1 cm. above the bottom of the buret. Thus, a differential pressure of less than 0.1 mm. of mercury is needed to force gas into the buret.

[1] *Ann. Repts. on Progress Chem. (Chem. Soc. London)*, **45**, 122–3 (1948).

[2] ARNOLD, R. C., A. P. LIEN, AND R. M. ALM, *J. Am. Chem. Soc.*, **72**, 731–3 (1950).

The buret is filled with water (preferably at room temperature) by shutting off the overflow, connecting an aspirator to the top, and running water into the 4-liter beaker at approximately the same rate as it rises. When the water has risen into the stopcock, the stopcock is closed. The buret is then ready for use after the overflow at the bottom has been opened.

The gas evolved from the reaction being studied is led, by means of 8-mm. glass tubing, through a cold trap (dry ice and acetone or methanol) into the bottom of the buret. When the evolution of the gas is complete, the number of mols of gas collected can be calculated from the following formula:

$$n = \frac{(P_{atm.} - P_{head} - P_{vap.})V}{RT}$$

where $P_{atm.}$ is the barometric pressure, P_{head} is the pressure due to the height of water above overflow, $P_{vap.}$ is the vapor pressure of water, and V is the volume of gas (obtained from the calibration table).

AN AMMONIUM DICHROMATE "VOLCANO"

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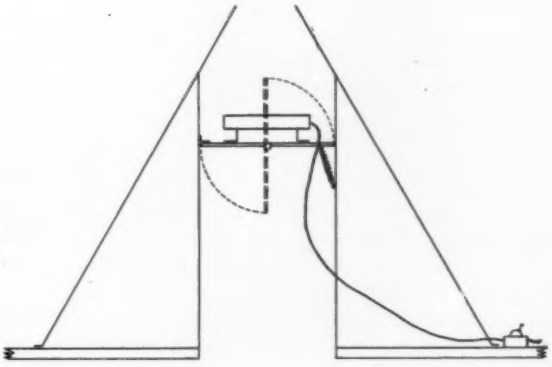
A POPULAR feature of several recent science exhibits at Huntingdon College has been the ammonium dichromate "volcanoes." These volcanoes have been made by students in general chemistry, and, with improvements from one year to the next, have evolved into the one described here.

To make the volcano erupt, about 10 g. of ammonium dichromate is poured in at the top and a switch at the base is thrown. At first a small amount of smoke comes out; then sparks and hot chromic oxide come pouring out. When the reaction is over, a small handle on the side is turned and the residual chromic oxide is dumped through the bottom of the volcano into a waste can below, and it is ready to erupt again.

The figure shows the general construction. Inside, about $2\frac{1}{2}$ in. from the top, is an iron disc to which is bolted the heating element. A 20-watt, nonvariable, 2500-ohm, vitreous resistor, of the sort available at a radio-supply store, serves as the heating element and is connected to a 110-volt source through a line out of the bottom of the volcano. An "on-off" switch is included in this line. Across the center of the disc is fastened a piece of stiff iron wire. This wire holds the disc in place, and it extends out through one side of the volcano to serve as a handle to rotate the disc through 90 degrees. A small spring is bolted to the



under side of the disc near one edge and brings the disc back to a horizontal position after dumping. Bolts, rather than solder, are used because of the heat generated. The heating element is well insulated from the reaction disc, so as to decrease the chances of electrical shock. The reaction chamber is a cylinder 3 in. in diameter made from a tin can.

The exterior of the volcano—a cone-shaped metal mantle—is covered with papier-maché and, near the top, asbestos pulp. The volcano is permanently mounted on a bridge table, and is about 14 in. high and 12 in. across at the base. The diameter of the opening at the top is $1\frac{1}{4}$ in.

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THE MUSPRATTS AND THE GAMBLES—PIONEERS IN ENGLAND'S ALKALI INDUSTRY[1]

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DURING the nineteenth century Great Britain attained a leading position in the alkali industry. Among the pioneers who contributed to English leadership were members of two remarkable Irish families, the Muspratts and the Gambles.

The first Muspratt of scientific interest was James Muspratt, who was born in Dublin, Ireland, on August 12, 1793.[2] He studied at a commercial school in the city and showed ability that might have earned him distinction had he been able to afford to go to college. But his parents were poor and could not afford this.

James Muspratt

At the age of 14, he quit school and was apprenticed to a Mr. Mitcheltree, a wholesale druggist, with whom he remained for four years.[3]

Muspratt found pharmacy interesting, for in those days it still had some of the wonder and mystery of old-time alchemy. There were, of course, no patent or ready prepared medicines or pills, and the pharmacist made up all his preparations from old herbal formulas, old mixtures handed down from generation to generation. He mixed his ingredients by hand in crude and old fashioned apparatus. Textbooks on pharmacy were few and far between, but whatever books Muspratt came across during his apprenticeship he read avidly, and he occupied himself in his spare moments by trying out chemical experiments.

When he was 18 years old Muspratt lost both his parents. Soon afterwards he quarreled with the old pharmacist and found himself out of work. He had no money, for what money his parents had left to him was tied up in legal wrangles. He joined the British army, and we next hear of him with the Duke of Wellington's forces at the Battle of Salamanca and entering Madrid with the British forces in 1812.

As the tide of war turned, Muspratt soon found himself in trouble. When large French forces were sent to Spain to counterattack the English forces Wellington retreated hurriedly from Madrid. Left behind in the haste, and ill with fever, was Muspratt. However, with the French only a few miles away, he decided to follow. Traveling on foot he performed the remarkable feat of covering 100 miles in two days and rejoining the main body of the army.

Muspratt later enlisted as a midshipman in the British Navy, but, though promoted to the rank of second officer, he disliked naval discipline and deserted when his ship anchored off Swansea, Wales. On returning to Ireland he found that the legal difficulties about his inheritance had been cleared up and a small capital sum was at his disposal. He looked around to see how best to make use of his money and decided to open a chemical plant, which must have been something of a venturous decision in those days.

He began by making hydrochloric acid but later, with a partner named Abbott, turned to the making of prussiate of potash also. He became a man of affairs in Dublin and a figure of note in the social and literary circles. His friends included Samuel Lover, the writer, and James Sheridan Knowles, the dramatist, after whom his son was named James Sheridan Muspratt.[4]

Prospects for chemical industry appeared better in England than in Ireland, so in 1822 he transferred his plant to a site in Liverpool. The new Leblanc proc-

[1] Presented before the Division of the History of Chemistry at the 119th Meeting of the American Chemical Society, Boston, April, 1951.

[2] HARTOG, P. J., "Dictionary of National Biography," Oxford University Press, London, 1909, Vol. 12, p. 1331. HAYNES, W., "This Chemical Age," Van Nostrand Co., Inc., New York, 1945, p. 26.

[3] ALLEN, J., "Some Founders of the Chemical Industry," 1906, p. 70.

[4] *Ibid.*, p. 76.

ess for making soda attracted his attention and he was quick to appreciate its commercial possibilities in view of the lifting at that time of the exorbitant duty of £30 a ton on salt.

In an old glassworks which he purchased on the banks of the Mersey river Muspratt began to operate a Leblanc plant in 1823. But the trouble at first was that no one would buy the soda he made by the new method. The soapmakers found the new soda to be very different to the barilla and kelp they were accustomed to and, though in fact it was a stronger and a purer product, they were suspicious of it, and preferred to purchase the better known materials even at higher cost.

Muspratt had to give away free large quantities of his product to overcome the prejudice[5] and, to prevent attempts at sabotaging his product, he had himself to superintend the making of soap from it, and so convince the soapmakers of its worth.[6] Of course, he eventually succeeded in this, and in order to satisfy the great demand the crude soda had to be delivered to the buyers straight from the furnaces, hardly cool enough to be handled.

His son, Edmund K. Muspratt, relates[7] that at first only "black ash" (the crude product of fluxing the materials in the furnace) was made, which tested 24 per cent Na_2O. This was a comparatively good product and was willingly taken by the soapmakers. But when it was found that the material lost strength on lying in the air—due to oxidation of calcium sulfide to sulfate and the action of the calcium sulfate on sodium carbonate—the further step was taken of treating the black ash with water and converting the solution by evaporation and calcination to white soda ash (as the French had done from the beginning).

Muspratt's plant prospered and, except for a few small works on the Tyne, his installation remained the only one in England for six years. As matters improved, Muspratt went into partnership with another chemist of Irish birth, namely Josias Gamble.

Gamble was born at Graan, near Enniskillen, county Fermanagh, and was of Presbyterian upbringing.[8] He decided to become a minister and took his master's degree in arts at Glasgow in 1797. Two years later he was ordained minister in his native Enniskillen and, after some service there, decided to go abroad as a missionary. It was the usual thing in those days for intending missionaries to take out a course of medical lectures in the Royal Academical Institution in Belfast to fit them better for the work.

The medical course included a series of lectures in chemistry, and so interested did Gamble become in chemistry as a result that when he returned home on vacation to Enniskillen he spent his time in what must have seemed an unusual occupation for a man of the cloth. He occupied himself in preparing bleaching solutions for linen made locally on the hand-loom. From the waste products of his bleach preparations he made Glauber salts, for which he found a ready market.

Josias Christopher Gamble

Apparently, Gamble abandoned his missionary ideas at this stage in favor of chemical industry, for shortly afterwards he spent some time operating a small bleaching plant in county Monaghan. Tennant had lately patented the process of absorbing chlorine with slaked lime and Gamble operated the process in his plant. Then he moved to Dublin and, while practicing intermittently as a minister, opened a larger plant on the banks of the river Liffey at Islandbridge above the city.

As well as bleach, he prepared the sulfuric acid needed for his process. In his acid plant he made use of new and improved techniques and was, for example, the first to adopt the French method of working vitriol chambers with a constant draft through them. It is said that he was so anxious to keep his methods a secret that, when making alterations in the plant, he carried out the work himself at night.

Before long he was also making alum, using as his raw materials potash from the brimstone ovens and pipeclay imported from Poole in England.

Like Muspratt, Gamble eventually decided to move to England where prospects for expansion seemed greater. He went to St. Helen's, met Muspratt, and decided to go into partnership with him.

Though commercially the soda process as operated by these two pioneers seemed headed for success, the acid released from the plant caused great damage to crops and pastures for miles around and, before long,

[5] LUNGE, GEORGE, "Manufacture of Sulphuric Acid and Alkali," 3rd ed., D. Van Nostrand Co., Inc., New York, 1909, Vol. II, Part II, p. 506.
[6] *J. Chem. Soc.*, 1871, 620.
[7] *J. Soc. Chem. Ind.*, 1886, 409.
[8] ALLEN, J., *op cit.*, p. 43.

various legal actions were brought against them either to mitigate the nuisance or to close down.

Muspratt and Gamble thought at first that if they built a very tall chimney it would carry the acid fumes to such a great height that by the time they came to earth they would be too dilute to do any damage. The chimney was built but the fumes were as damaging as ever. The trouble was that the humidity was so great that, despite the chimney height, the acid condensed at once on leaving the top and fell to the ground, destroying vegetation just as before.

The pair tried other expedients and in 1836 devised a coke condenser arrangement to remove the acid fumes and so prevent the fumes from reaching the atmosphere at all. Muspratt did not think much of this scheme, particularly after the various earlier abortive experiments, and could not see how the condenser could get rid of the great volume of fumes involved. He remarked "Sure all the waters of Ballyshannon itself would not suffice to condense the acid I make."[9]

But the condenser arrangement did prove a qualified success and the new industry developed rapidly. Soon afterwards Gamble branched off independently and later operated plants in various English towns. Muspratt expanded into new plants all over Lancashire. One small town where he opened a plant expanded so rapidly as a result that its population rose from 3000 to almost 25,000 people in a 20-year period.

A later development in the alkali industry associated with the names of these pioneers was the evolution of the plus-pressure furnace. This was the name of a type of salt-cake furnace adopted widely since 1876, in which the escape of acid-gas through leaks in the muffle into the fire-flues (without passing through the condensing apparatus) was ingeniously avoided. This was done by making the draft within the muffle stronger than in the fire-flues surrounding it. If any leaks existed in the muffle, the acid-gas would not escape into the flues. Instead, a little fire-gas would enter into the muffle, which was not considered harmful and which in any case stopped all complaints about the escape of acid from the plant chimney.[10]

Muspratt's method of achieving such a plus-pressure was by dispensing with the chimney-draft, and, instead, forcing air underneath the grate, and also admitting additional air in other parts of the furnace for burning the carbon monoxide.[11]

In Gamble's furnace a blind roaster was fired by means of gas producers; the air required for combustion was heated by passing through a system of iron pipes which, on their part, were heated from the outside by the heat of the furnace-fire. The escape of acid vapors through any chinks from the muffle into the fire-flues was avoided by adjusting the dampers so that the gas in the generator was kept at a slightly higher pressure than the gas inside the muffle.

Though both Gamble and Muspratt lived for many years in England, they always maintained a keen interest in Irish affairs. Gamble was of liberal outlook, and is reputed to have been a member of the secret revolutionary "United Irishmen" when he lived in Dublin. During the abortive rebellion in 1798 he sheltered and hid a number of the important leaders of the movement in his home.

Muspratt's interest in Irish affairs was illustrated by his action regarding the use of Irish-mined pyrites. About the year 1832 the government of Naples in Italy raised the price of native sulfur to a very high figure. The result was that Muspratt and other English manufacturers turned to Irish deposits of iron pyrites in county Wicklow. Until the Neapolitan price was again lowered trade was brisk in the Irish ore and over 100,000 tons a year were exported.[12] It is of interest to note that these deposits were not again considered until the time of World War II, when a scheme was on foot to recover sulfur from them for eventual use in the making of superphosphate fertilizer.

When Liebig was experimenting on the refertilization of soils Muspratt cooperated with him in the making of various chemical fertilizers. The venture was not a success at the time, for the raw materials were too costly. Yet the work was a forerunner of later large-scale developments in the chemical fertilizer industry.

Throughout his business life James Muspratt believed in free trade. He did not wish for protective tariffs for his industries, but held the view that if his products were of high enough quality they could stand on their own feet against foreign products. In his later years he made extended visits to the European continent, and often visited with Liebig in Giessen. In retiring from an active part in his plants he became interested in education and was one of those responsible for developing the Mechanical Institute of Liverpool. This school made vocational and technical training available at low cost to workers in the area.

James Muspratt died at the age of ninety in Liverpool. His had been a busy and active life, for the chemical manufacturer had many problems to contend with in those days. There was always the chance of new discoveries and inventions coming on the market and perhaps making a whole plant or series of installations obsolete overnight. When not facing legal action for damage caused by effluents from his plants, the manufacturer was fully occupied in training workers in skilled operations and in supervising experiments to improve yields and better his products.

It has been said of James Muspratt that he will be remembered not so much as an original inventor or as a chemist, "but as a man of unusual energy, of keen perception, of indomitable perseverance, and of far-seeing enterprise, and such men have been large con-

[9] Ballyshannon, on the river Erne, is now the site of a large Irish hydro power plant.

[10] LUNGE, GEORGE, "Manufacture of Sulphuric Acid and Alkali," 3rd ed., Gurney & Jackson, London, Vol. II, Part I, 1909, p. 170.

[11] British Patent, May 4, 1877.

[12] REILLY, D., "Sir Robert Kane," Cork University Press, Ireland, 1942, p. 38.

tributors to the greatness of the countries in which they worked.[13]

Apart from the work already described, two other important advances in the alkali industry were brought about by Josias Gamble. In the first place, he suggested the use of boat-pans in the evaporation of alkali plant solutions for solids recovery. Various open-pan heating methods were first developed in France, Germany, and England. However, Gamble put forward the suggestion that instead of using flat-bottomed pans or pans provided with gutters, pans shaped like boats should be used. The idea of such pans was to allow the salt to drop to the bottom as the solution became more concentrated, heat being applied only to the sides of the pans. Pans of this type were later adopted widely in England.[14]

Gamble's second contribution was in the field of solid caustic production. A new epoch dated from Gossage's patent of May 18, 1853. Essentially, Gossage's process first freed soda-ash liquor from iron sulfide by air oxidation, and then treated it gradually with a solution of bleaching powder to convert the thiosulfate into sulfite or sulfate and concentrated it until nearly all the sodium carbonate and ferrocyanide had been separated as salts. The remaining solution was then concentrated to yield solid caustic.[15] According to Davis,[16] Gamble was the first to carry out Gossage's process on a large scale. The industry expanded rapidly in the South Lancashire area, which eventually became the English center of such manufacture.

The ability of early pioneers to develop uses for waste products was well illustrated by Gamble in describing uses for hydrogen sulfide at that time.[17] While it was understandable that pure hydrogen sulfide was readily burned for conversion to sulfuric acid, Gamble mentioned that even dilute sulfide—as obtained for instance in making ammonium sulfate, as well as in various attempts at decomposing alkali wastes with carbon dioxide—can under favorable conditions be converted into sulfuric acid in ordinary vitriol chambers. He described how this was done as early as 1839 in at least four Lancashire works.

When Josias Gamble retired from business his work was carried on by his son, David Gamble, who was also a chemist, but better known as an educationalist.[18]

Muspratt, too, was succeeded by his sons, all four of whom were chemists. Notable among these were Edmund Knowles Muspratt, founder of the Muspratt Physical Chemistry Laboratory, and James Sheridan Muspratt.

The latter was born in Dublin on March 8, 1821. He inherited his father's interest in chemistry and, when only 13 years of age, enrolled in Glasgow University, later going on to London University. He became chemist to a plant in Manchester at the age of 17 and in the same year published his first scientific paper, which dealt with chloride of lime.

His father's industrial plants were then at their peak and, after young Muspratt had made an unsuccessful attempt at embarking on a business career, his father advised him to spend more time in the study of chemistry.[19] As a result, he went to work for two years in Liebig's laboratory in Giessen. He published a detailed paper on the sulfites, which contained much that was new on their chemical nature and which was widely translated. The work won him his Ph.D. degree while still but nineteen years old.

On his return to England he read a paper on the work to the British Association for the Advancement of Science. Later he spent some time at work with Hofmann, and discovered and described a number of important organic intermediates which were later to be of significance in the dye industry. With Hofmann, he also isolated a new acid from cloves. In later years he was the founder of the Liverpool College of Chemistry, which proved to be the training ground of many notable English chemists.

But the most striking contribution to scientific progress made by James Sheridan Muspratt was in his massive two-volume work of 2000 pages, his "Dictionary of Applied Chemistry." The work appeared first in monthly parts and was subsequently published in book form in Glasgow and London in 1860. It was dedicated jointly to the French chemist, J. B. Dumas, and to Sir Robert Kane, noted Irish industrial chemist.[20] Muspratt wrote: "To whom could I with greater propriety inscribe this work than to my distinguished friends and colleagues; both of whom have contributed the most valuable assistance to the Arts and Manufacturers of their respective countries, as well as to the world in general."

By way of a conclusion to this account of some Irish pioneers in the British alkali industry, some mention might be made of a certain Michael Murphy from county Clare in the west of Ireland. In the preface to Muspratt's dictionary, a brief reference is made to the work done by Murphy in helping to compile it.

Murphy's story is a remarkable one of self-advancement. He began as a youth without any special schooling or training. He travelled on foot from his native town to Dublin, a distance of some 150 miles, and worked his passage on a boat to Liverpool. In that city he got a small job in one of the Muspratt plants.

He studied privately, and gradually advanced in status, with the result that when the College of Chemistry was opened in Liverpool, he took full charge of it for a number of years. Almost the whole work of editing the dictionary fell on Murphy's shoulders, and it was related that he worked 16 hours a day for two full years on the task. In 1877 he built his own factory in Liverpool and there made oxide of cobalt.

[13] ALLEN, J., *op. cit.*, p. 100.
[14] LUNGE, GEORGE, *op. cit.*, Van Nostrand Co., Inc., New York, 1909, Vol. III, Part II, p. 697.
[15] *Ibid.*, p. 790.
[16] *J. Soc. Chem. Ind.*, **1886**, 351.
[17] *J. Soc. Arts*, **1882**, 735.
[18] *J. Chem. Soc.*, **1908**, 2279.
[19] "Memoir of J. S. Muspratt," preface to MUSPRATT, J. S., "Dictionary of Applied Chemistry," London, **1860**, p. i.
[20] REILLY, D., *op. cit.*, p. 22.

Proceedings of the

PACIFIC SOUTHWEST ASSOCIATION OF CHEMISTRY TEACHERS

LECTURE DEMONSTRATIONS WITH THE NEWER TYPES OF PLASTICS[1]

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SINCE the war there have been rapid and spectacular developments in the field of plastics. Many of these new materials are suited for use at the lecture table and can be quickly demonstrated with professional results provided intermediate molding powders or prepared liquid resins are used.

The trade names and commercial sources of such polymers are included in this article, which has been written to serve as an outline that will supply the teacher with pertinent information regarding the properties, identification, uses, and demonstration preparation of typical resinoids. The prices quoted are for commercial quantities and are subject to change. They have been listed to assist the demonstrator in estimating his expenditures.

The resins to be described include Thiokol, polyvinyl chloride, phenol formaldehyde, styrene polyesters, ethyl cellulose, and polymethyl methacrylate. They have been organized into thermosetting and thermoplastic types which in turn are classified under the general headings of synthetic elastomers and plastomers.

SYNTHETIC ELASTOMERS

Thiokol (Thermosetting)

The need for synthetic rubber materials, called synthetic elastomers, has spurred the development of several types including Neoprene, Thiokol, styrene-butadiene copolymers, acrylo-nitrile and polyvinyl chloride polymers. Of these, polyvinyl chloride and Thiokol may be conveniently used for demonstration. The latter, a solvent resisting compound, will be described first. Its name, Thiokol, is used in the trade to cover the class of polymers derived from ethylene dichloride and sodium polysulfide.

Thiokol is used commercially for weather stripping and as a caulking compound. In the latter application it can be slushed around inside an airplane wing,[1] sealing it and converting it into a gasoline tank.

Equipment. Beaker, 2 broad-bladed spatulas, balance, 100 g. PK 1123[2] (a dark viscous liquid low-polymer Thiokol resin), 20 g. lead peroxide to serve as a polymerization catalyst.

Directions (time, 10 min.). Mix the resin and catalyst together in the beaker with a spatula. Scrape the sticky mass from this spatula onto another one. Remove the product from the spatula and work into a ball with the hands. After about five minutes of kneading, the brown colored ball will lose its sticky quality and take on elastic properties which can be dramatically demonstrated by bouncing it on the floor.

Thiokol has a slight but persistent obnoxious odor. Since PbO_2 is poisonous, hands and utensils should be kept well cleaned with soap and water. The sticky mixture may be removed with acetone.

Identification. Upon heating, Thiokol decomposes to give the odor of rotten eggs. If the fumes are passed through barium chloride solution a precipitate will form.

Discussion. At room temperature the lead dioxide catalyzes the further polymerization of the low-polymer Thiokol to form a solid which at this temperature is an elastomer or rubbery-like substance. The mechanism of the polymerization involves chain growth and cross linking. The polymers are said to be elastomers when they exhibit elastic behavior, *i. e.*, elongation and recovery. During catalysis the lead atom is believed to serve as a connecting link between the sulfide end of one chain and that of a similar chain. The lead finally splits out of the combined chains as PbS, leaving the two chain units connected with each other by a sulfur atom. Water as well as PbS is formed as a by-product of catalysis.

[1] Based on material presented before the Southern California Section of the Association on May 25, 1951, at Occidental College.

[2] Plas-Kem Corp., 100 W. Alameda Ave., Burbank, California. (About $2 per lb.) *Note:* All other resins mentioned in this article may be obtained from Plas-Kem Corp. as well as from the manufacturers listed. All prices given are approximate ones.

Polyvinyl Chloride (Thermoplastic)

This compound is potentially the lowest-cost plastic of the vinyl resins, which include polyvinyl acetate, polyvinyl butyral, polyvinyl chloride-acetate copolymer, polyvinyl formal, and polyvinylidene chloride. It may be purchased in the form of a coarse white powder, Geon 101,[3] or as an extremely fine white powder, Geon 121.[3] The Geons are high polymers which need merely to be plasticized to form a usable plastic.

The resin is used wherever the requirements are toughness, flexibility, high dielectric strength, and resistance to water, alcohol, and acid. Geon 101 is employed in the manufacture of extruded garden hose, laboratory tubing, electrical insulation, calendered film, flexible molded goods, and linings for acid plating tanks. Geon 121, which provides a more uniform mix, is used in making dolls, rainboots, simulated leather, and flexible molds. By impregnating canvas with this resin, an acid- and fungus-resistant tarpaulin is formed.

Geon 101

Equipment. Small pyrex sauce pan with handle, hot plate, shallow metal or enamel pan containing a few flat objects such as keys and coins, six-inch spatula, balance, 50 g. Geon 101 resin, 200 g. dioctyl phthalate (a clear liquid plasticizer of low volatility).

Directions (time, 15 to 20 min.). With the spatula, mix the resin and plasticizer together in a pyrex sauce pan over a hot plate at about 175°C. (Avoid spilling dioctyl phthalate since it will dissolve lacquer on desks or tables.) Heat the mixture until it is melted to a fluid state, stirring continuously to prevent burning. Pour the amber or reddish-colored sirup into a shallow dish containing the keys and coins. When solidified pull out the translucent rubber-like slab and remove the articles. Call attention to the fine detail of the imprints left by the articles upon the surface of the plastic. This procedure can be used to prepare molds for lower-temperature-setting materials such as the phenolic resin to be described in the section on synthetic plastomers. With less plasticizer the product would be dryer and would be suitable for molding by pressure.

Identification. The plastic melts when heated. It is flame resistant, however, for it will stop burning when removed from a flame. The presence of chlorine may be detected by the Beilstein test.

Discussion. The resinous mixture or plastisol prepared in this experiment is a colloidal dispersion of the resin in the plasticizer, the latter serving as a peptizing agent. Heating to 175°C. hastens the conversion of the sol to the gel desired as the end product.

Geon 121

Equipment. Beaker, hot plate, 150°C. drying oven, balance, cavity mold[4] of doll's head or of some other object, basin of water for cooling the hot mold, broad-bladed spatula or preferably a $^1/_8$ to $^1/_4$-inch metal rod, such as a ring stand rod, to serve as a mandrel, 50 g. Geon 121 resin, 50 g. dioctyl phthalate plasticizer.

Directions (time, 30 to 45 min.). With the spatula, mix the resin and plasticizer in the beaker. Heat the mandrel and dip it into the mixture or plastisol. If the mandrel is at the proper temperature the material will jell on the hot surface. The longer the mandrel is left in the mixture the more of the plastisol it will pick up. For thicker coatings the mandrel may be reheated and redipped. After the mandrel is sufficiently coated remove it from the plastisol and heat over the hot plate, rotating the mandrel until the plastic is set. The rubbery resin may then be slipped off the mandrel and onto a finger as a "finger stall." This part of the experiment takes about 10 minutes and demonstrates the physical changes that occur in the plastic if prepared at the right temperature.

To demonstrate the commercial application of this material either the mixture prepared above or a commercially prepared plastisol[5] can be used. The casting material prepared above, however, will be softer and less durable in appearance than the commercially prepared plastisol in which there can be a higher concentration of the resin due to the efficiency of mechanical mixing.

Pour the plastisol into a mold which has previously been heated for about 10 minutes at 150°C. Swish the material around in order to coat all the interior of the mold, and pour slowly to avoid the formation of air bubbles. The plastisol immediately in contact with the hot inner surface of the mold will jell. The excess unjelled material may then be poured out and the plastic-coated mold set on the hot plate or in an oven at 150°C. to bake for approximately 15 minutes. After baking, cool the hot mold by immersing in cold water for about a minute to prevent distortion. Then the elastic fleshy-colored replica may be pealed out.

Identification. The demonstrations performed with Geon 101 are applicable here. A microscopic examination of the two Geons will exhibit a noticeable difference in the size of the particles.

SYNTHETIC PLASTOMERS

Phenol Formaldehyde (Thermosetting)

This is the most widely used thermosetting resin and is one of the pioneer resinoids, universally recognized under the trade name, bakelite. Low polymers of phenolic resin are available in liquid or solid form.[6] A fabric base laminated sheet impregnated with phenolic resin may be heated over a hot plate and bent to demonstrate the post-forming technique. Whereas phenolic molding materials contain wood flour or as-

[3] B. F. Goodrich Chemical Co., Cleveland, Ohio. (Geon 101: 44¢ per lb. Geon 121: 46¢ per lb.)

[4] An ordinary metal crucible may be used as a mold. A lead mold may be obtained from Plas-Kem Corp.

[5] Plas-Kem Corp. ($1 per lb.)

[6] Bakelite Corp., New York, N. Y. These are basic resins. More suitable for demonstration purposes is the specially prepared resin referred to in the following experiment as Phenolic Casting Resin.

bestos fillers other molding materials do not. For the experiment to be described, however, a phenolic casting resin is employed which contains no filler.

Phenolic resin is used commercially in compression-molded radio and television cabinets, washing machine agitators, flat-iron handles, cast statuary, switch and electrical components, insulating varnishes, plywood adhesives, and laminated table tops. The post-forming technique of handling laminated materials is widely used in the aircraft industry and in the manufacture of ammunition boxes.

Equipment. Beaker, stirring rod, 100°C. oven, mold[7] or expendable beaker, balance, 85 g. Phenolic Casting Resin[8] (an amber viscous liquid), 15 g. acid catalyst.[9]

Directions (time, 30 min.). Thoroughly mix the resin and catalyst together in the beaker and pour the mixture into the mold. Bake the preparation in a 100°C. oven for 20 minutes. The hard amber-colored casting can be removed forcibly from the mold or by breaking the expendable beaker.

Identification. The heat resistance of the plastic is clearly illustrated by its failure to soften or burn in a flame. When exposed to the flame, however, the odor of formaldehyde is detectable. This is dissipated almost instantly and the more persistent odor of phenol may then be smelled. The plastic does not dissolve in any solvent.

This experiment illustrates the formation of a cross-linked, thermosetting plastic.

Styrene Polyesters (Thermosetting)

A typical styrene polyester such as Paraplex P-43[10] is composed of a mixture of liquid monomeric styrene and a polyester made from diethylene glycol and maleic anhydride. It is used in electrical "potting" compounds, *i.e.*, as a casting around a coil or electrical circuit to hold the components rigidly in place. It is employed as a laminating resin with glass mat and glass fabric in the production of boat hulls, airplane fuselage tail and wing sections, ventilating and heating ducts, and prefabricated wall sections. It is also used as a resin binder in rocket propellent charges.

Equipment. Beaker, stirring rod, 100°C. oven, flexible casting mold or expendable beaker, 100 g. resin, Paraplex P-43 (a clear yellow liquid), 5 g. benzoyl per oxide, a solid catalyst.

Directions (time, 45 min.). Stir the resin and catalyst together in the beaker until the peroxide is dissolved or well dispersed. Bake the mixture in a mold in a 100°C. oven for 15 to 30 minutes until the substance is solidified. The casting is a yellow, clear, tough plastic. Benzoyl peroxide is an explosive and customary precautions must be observed in its use. Do not grind or heat it other than as directed.

Identification. When burned the plastic emits a black sooty flame and a sweet odor, the latter indicating the presence of esters; the former, the presence of styrene. It is distinguished from polystyrene in that it does not melt on heating or dissolve in solvents.

Discussion. The benzoyl peroxide is frequently used as an oxidation catalyst in polymerization processes. In this experiment it acts as a chain initiator. First it decomposes to form a free radical, then it adds to an unsaturated point on the styrene molecule or on the unsaturated polyester molecule to form a new free radical. The chain thus started adds styrene or polyester until terminated by another free radical or another chain.

Ethyl Cellulose (Thermoplastic)

This is made by treating about two-thirds of the hydroxyl groups on the cellulose molecule with sodium hydroxide and then etherifying with ethyl chloride. It is available as a white granular powder.[11, 12] It is used as a strippable coating in packaging articles for overseas shipment. Antioxidants may be added. Because of its high impact strength it is employed for jigs and forming dies, screw driver handles, and flashlight cases.

Equipment. Stirring rod, pyrex sauce pan with handle, hot plate, tapered crucible for a mold, 100 g. ethyl cellulose resin C-10 grade or N-10 grade, 100 g. castor oil as plasticizer.

Directions (time, 15 min.). While heating, carefully mix the ingredients in the sauce pan on the hot plate, stirring continuously until the mixture is well liquefied. Pour the sirup into a suitable mold (greased to prevent sticking) and allow it to cool. The casting is tough and flexible, although not elastic.

Identification. Ethyl cellulose is unusually stable toward heat. Hold a sample in a flame and catch the drippings in water. The drippings will still be tough and thermoplastic, whereas other cellulose resins would decompose under similar conditions.

Polymethyl Methacrylate (Thermoplastic)

This thermoplastic polymer is derived from methyl methacrylate liquid monomer, which is the methyl ester of the methyl substituted acrylic acid. The polymer is the most water-clear plastic available and can be obtained in sheet form from E. I. du Pont de Nemours and Co. or Rohm and Haas Co. Du Pont sells it under the trade name, Lucite, while Rohm and Haas call their product Plexiglass. It is used in the manufacture of cockpit enclosures, nonbreakable windows, street light globes, fluorescent lighting fixtures, and dentures. It is also employed to imbed biological specimens for display and preservation.

[7] The mold may be prepared as a flexible casting by the procedure used in the Geon 101 experiment since the latter is stable at 100°C. This demonstration resin-made mold may be used or a commercial mold may be obtained from Plas-Kem Corp. ($5 per mold.)

[8] Lebec Chemical Co., 6000 So. Boyle Ave., Los Angeles, California. (60¢ per lb.)

[9] 10 per cent HCl may be used. It is recommended, however, that a specially prepared acid catalyst be used. Such an acid is supplied with the Phenolic Casting Resin mentioned above.

[10] Rohm and Haas Co., Inc., Philadelphia, Pa. (60¢ per lb.)

[11] Dow Chemical Co., Midland, Michigan. (C-10 grade, 60¢ per lb.)

[12] Hercules Powder Co., Wilmington, Delaware. (N-10 grade, 60¢ per lb.)

Shaping

Equipment. Hot plate, a bowl, a sheet of Lucite or Plexiglass.

Directions (time, 10 min.). Heat a sheet of the thermoplastic over the hot plate. While hot and flexible stretch it over an inverted bowl. Hold it in this position until it has set. The setting may be hastened by immersion in water. The "plastic memory" of Lucite may be illustrated by reheating the bowl-shaped sample over the hot plate until it softens when it will return to its original flat sheet form.

Imbedding Objects[13]

Equipment. Beaker, stirring rod, 80°C. drying oven, balance, a suitable mold such as a Petri dish or expendable beaker, an object to be imbedded such as a crystal or mineral sample, 100 g. pure methyl methacrylate,[14] 0.2 g. benzoyl peroxide catalyst.

Directions (time: mixing time, approximately 15 min.; curing time, approximately 1 hour for each 1/4-in. layer). Prepare a cold mix of the ingredients in a beaker by dissolving the benzoyl peroxide in the methyl methacrylate. Pour a 1/4-inch layer of the liquid into the mold containing the object to be imbedded and cure slowly in an oven at 80°C. Repeat this process, building up successive layers until the object is covered to the desired depth.

An alternate method is to prepolymerize the monomer to a heavy sirup by heating it in a beaker on a water bath over a hot plate and then pouring over the object to be imbedded and curing the entire mass at 80°C. in one operation.

The monomer is very flammable and develops exothermic heat on curing. Therefore, if the monomer is used, only small masses should be cured at a time or bubbles will develop from the heat of polymerization.

Identification. Upon heating the plastic the characteristic sweet odor of the monomer, methyl methacrylate, is easily distinguishable. The plastic resinoid decomposes in a gas flame to produce the monomer which burns with a smooth blue flame. By heating pieces of the plastic in a flask the polymer chains are broken to yield the monomer which can be recovered by distillation, demonstrating the reversal of polymerization.

Discussion. The monomer or low polymer is converted by the benzoyl peroxide catalyst to the solid high polymer form. Since this product is not cross linked, it is thermoplastic and softens around the boiling point of water. In this experiment the preparation of a cold mix of monomer and catalyst was followed by a heat cure. A cold cure is possible but very slow.

[13] Special preparation is necessary to imbed biological specimens.

[14] The commercial monomer may be obtained from du Pont. ($1 per lb.) *Note:* A tertiary butyl catechol or hydroquinone stabilizer is normally present in the commercial monomer and must be removed by vacuum distillation or by washing the monomer with alkali and drying.

A LECTURE REPRESENTATION OF OXIDATION-REDUCTION

R. T. SANDERSON
State University of Iowa, Iowa City, Iowa

MANY students of elementary chemistry have difficulty in understanding the concept of oxidation-reduction, even in its simplest application to reactions which actually form ions. In particular, it seems hard for them to comprehend that by the process of acting as an oxidizing agent, an atom becomes a reducing agent, and *vice versa*, and that whether or not an atom or ion will act as a reducing agent or oxidizing agent or neither depends on *relative* attractions for electrons.

A very helpful demonstration of oxidation-reduction by electron transfer can be presented with the aid of a few permanent magnets, of progressively increasing strengths, to represent atoms of oxidizing agents, and an iron object to represent an electron. For example, suppose one has four magnets, differing in strength, the strongest representing an atom of fluorine, the next strongest representing an atom of chlorine, the next an atom of bromine, and the weakest an atom of iodine. One may first demonstrate that each atom is capable of taking on an electron, and thus acting as an oxidizing agent, by showing that each of the magnets can pick up a nail representing the electron. Pointing out that any atom which has taken on an electron can give it up again, thus acting as a reducing agent, one can then demonstrate that the iodine magnet, after taking on an electron to become an iodide ion, can give up the electron to the bromine, chlorine, and fluorine magnets. Similarly, one can show that the fluorine and chlorine magnets but not the iodine magnet can take an electron away from the bromine magnet, and that only the fluorine magnet can take the electron away from the chlorine magnet. In such a manner the relative oxidizing strengths of the halogen atoms and the relative reducing strengths of the halide ions can be demonstrated by an analogy which is easy for the student to understand and to extend to oxidation-reduction reactions in general.

Magnets can easily be adjusted to the desired relative strengths by gluing a very few thicknesses of thin paper or Scotch tape over the ends.

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

THIRTEENTH SUMMER CONFERENCE

With Donald C. Gregg of the University of Vermont, Burlington, Vermont, and William S. Huber of the Rhode Island School of Design, Providence, Rhode Island, as co-chairmen, the Thirteenth Summer Conference of the New England Association of Chemistry Teachers met at the University of Rhode Island, Kingston, Rhode Island, August 20–25, 1951. President Carl R. Woodward of the University of Rhode Island, who welcomed the conference to the campus, called attention to the fact that this was the first group to use the new chemistry building. It was later occupied by classes for the first time during September. The conference was similar to previous ones in the high quality of the speakers, the free and even heated discussion of papers and the relaxed enjoyment of the splendid facilities of the university and surrounding vacation areas. On the other hand, each conference is sufficiently different to maintain the interest of members and their families, many of whom return year after year.

The list of those who have attended all of the 13 Summer Conferences remains unchanged this year:

Mr. and Mrs. Grover Greenwood, Bridgeport, Connecticut.
Mr. and Mrs. S. Walter Hoyt, Belmont, Massachusetts.
Alfred R. Lincoln, Williamsett, Massachusetts.
Evelyn S. Murdock, Stonington, Connecticut.
Elsie S. Scott, East Northfield, Massachusetts.
John R. Suydam, Southboro, Massachusetts.
Elbert C. Weaver, Andover, Massachusetts.

Of the 204 registrants, almost exactly one-third were from outside New England. The 129 members attending came from the following areas: New England States, 96; New York, 15; Maryland, 6; Pennsylvania, 4; New Jersey, 3; Virginia, 2; and one each from Ohio, Indiana, and Quebec.

In addition to the papers presented by individual speakers from many colleges and several industries, two symposia were presented. The first was concerned with the Content of the General Chemistry Course, with Laurence S. Foster, Watertown Arsenal Laboratory, and Dorothy W. Gifford, Lincoln School, discussing the secondary-school course, and with

John E. Cavelti, Allegheny College, John A. Timm, Simmons College, and Roland M. Whittaker, Queens College, discussing various types of courses at the college freshman level. The second symposium, arranged by Elbert C. Weaver, Andover Academy, took the form of a workshop on Pupil Participation in Classroom Demonstrations. The Chemistry Department at the University of Rhode Island graciously opened the stockrooms to provide the teachers with chemicals and equipment for the demonstrations and the conferees performed both as demonstrators and as pupil participators. The most ingenious demonstration showed how a "neutron" (a free running half-back professor) could pass through atoms made up of whirling and rotating "electrons" (lady teachers) surrounding dense "nuclei" (left to the reader's imagination) without colliding with anything most of the time.

The Exhibit Committee, headed by George Deckey, Rhode Island School of Design, succeeded in securing numerous interesting collections of books, apparatus and literature. Ralph Di Mattia, Grover Cleveland School, Dorchester, Massachusetts, projected a fine collection of industrial motion pictures.

The social hours, following the evening meetings, were enlivened by a demonstration of parlor magic by Dean Woodward of the University of Rhode Island and a showing of colored lantern slides by NEACT President Helen W. Crawley of the Walnut Hill School, taken on a trip to the West Coast. At the social hour on Friday night, the William F. Ehrets of New York University, who had just returned from a year at the University of Hawaii, entertained the group. Mrs. Ehret and their daughter, Elizabeth, gave two native dances while Professor Ehret took the group on a trip around the Islands (in natural color).

The customary Wednesday picnic was a repetition of the successful one last year, featuring Rhode Island quohaug chowder engineered by Clinton S. Johnson and sweet corn grown and cooked by Bernard Jurale. Rhode Island lobsters from nearby Galilee were also provided.

MINUTES OF THE ADJOURNED ANNUAL MEETING, AUGUST 22, 1951

The meeting adjourned *sine die* on May 12, 1951, was reopened by the President, Helen W. Crawley, at 11 A.M.

The following report of the Treasurer, Carroll B. Gustafson, was accepted:

Balance on hand, 8/11/50	$1,126.70	
Income for the year 1950–51	2,075.30	
		$3,202.00
Expenditures, 1950–51		1,754.79
Balance on hand, 8/11/51		$1,447.21

The secretary reported for the Membership Committee the election of the following new members since the May meeting:

Edith N. Evans, Head of English Department and Teacher of Chemistry, Oxford School, Hartford, Connecticut.

William P. Galvin, Chemist, Watertown Arsenal Laboratory, Watertown, Massachusetts.

Thomas R. P. Gibb, Jr., 12 Hereford Road, Marblehead, Massachusetts.

Mrs. James E. Sague, Professor of Chemistry and Chairman of Department, Vassar College, Poughkeepsie, New York.

Robert E. Schaffrath, Instructor of Chemistry, New England College, Hillsboro, New Hampshire.

Morton Jay Zerder, In charge of Science, Walter Hervey Junior College, 15 West 63rd Street, New York 23, New York.

New members admitted in connection with the Summer Conference are:

Henry H. Baker, Jr., Professor of Chemistry, U. S. Naval Academy, Annapolis, Maryland.

W. Lawson Batty, Jr., Sales Representative, Mallinckrodt Chemical Works, New York, New York.

Mrs. William Brauner, 15 Benton Street, Wellesley 81, Massachusetts.

John E. Cavelti, Professor of Chemistry and Department Head, Allegheny College, Meadsville, Pennsylvania.

Paul F. Davis, Department of Science, Bellows Falls High School, Bellows Falls, Vermont.

Mrs. Alice Howe, Science Instructor, Concord High School, Concord, Massachusetts.

George R. Laurence, Box 863, Chatham, Massachusetts.

Harold A. Miner, Science Instructor, Northboro High School, Northboro, Massachusetts.

W. Donald Pennington, Associate Professor of Chemistry, U. S. Naval Academy, Annapolis, Maryland.

Theodore M. Peterson, Teacher of Chemistry, Jamestown High School, Jamestown, New York.

Ida B. Routh, Teacher of Chemistry, Bryant High School, Long Island City 3, New York.

Helen A. Sperry, Science Teacher, The Beard School, Orange, New Jersey.

Angela M. Trovato, Chemistry and Biology Teacher, Robinson Seminary, Exeter, New Hampshire.

Anna Vafiades, Laboratory Assistant in the Physical Sciences and General Science, Long Island City High School, New York.

Thelma G. Voss, Chemistry Teacher, Woodbury High School, Woodbury, New Jersey.

The Secretary reported that the organization had a membership of 522 on May 12, 1951, and that now the membership stands at 533.

Upon motion it was voted to extend the thanks of the Association to William S. Huber and Donald C. Gregg for their services as co-chairmen of the 13th Conference, to Clinton S. Johnson and Bernard Jurale for making the picnic a success, and to W. George Parks and the University of Rhode Island for facilitating the success of the Conference.

The President announced that Marco Scheer, Nashua High School, Nashua, New Hampshire, and William F. Ehret of New York University would act as Co-chairmen of the 14th Summer Conference, to be held at the University of Vermont, the third week in August, 1952. Dr. Ehret is to be Chairman of the Program Committee.

The places and dates for the five division meetings for the year 1951–52 were announced:

October 13, 1951, Manchester Central High, Manchester, New Hampshire.

December 8, 1951, Brookline High School, Brookline, Massachusetts.

February 9, 1952, Brown University, Providence, Rhode Island.

April 12, 1952, Deerfield Academy, Deerfield, Massachusetts.
May 10, 1952, College of the Holy Cross, Worcester, Massachusetts.

Mr. S. Walter Hoyt, Chairman of the Necrology Committee, offered the following resolution on the death of Gorham W. Harris, which was voted:

WHEREAS: Death has taken Gorham W. Harris, a member of the New England Association of Chemistry Teachers since 1921 and an honorary member since 1949.
BE IT RESOLVED: that our members gathered at their 13th Summer Conference at the University of Rhode Island do hereby testify to the outstanding character of the work of Professor Harris as a teacher of chemistry at Simmons College, at Radcliffe College, and at the Harvard Summer School, and his work as an incorporator and Executive Committee member of the Northeastern Section of the American Chemical Society, and
BE IT FURTHER RESOLVED: that the sympathy of the Association be tendered to his family, that a copy of these resolutions be sent to his wife and that a copy of these resolutions be entered upon the records of the Association.

LEALLYN B. CLAPP, *Secretary*

To the Editor:

In connection with the article by Mathews[1] on the construction of electric flask heaters, your readers may be interested to know of a somewhat similar method of construction which was described by the writer in 1949.[2] The chief points of difference are that the elements are wound on asbestos string which is in turn wound onto the inverted flask and plastered with asbestos lagging pulp, and secondly, in the place of cans ordinary flower pots are used. These have the advantage of being nonconducting and because of their tapered bases they can be fitted into the ordinary ring supports.

The method of construction is not limited to single flask heaters, but has also been used for a bath for eight Soxhlet units.

A limited number of reprints of the paper cited are available for any who care to write for them.

M. S. BUTLAND

NEWMARKET, AUCKLAND
NEW ZEALAND

To the Editor:

While teaching sophomore quantitative analysis during the past several years, I have noticed a peculiarity about the commonly used 50-ml. Exax burets. I have been wondering if others among your readers might either confirm or deny my observations.

Each year I have the students calibrate their burets. Student calibrations, especially at the beginning of the year, are notoriously unreliable. Nevertheless, a striking regularity is revealed from some hundreds of calibrations. The over-all corrections to the burets are *always* positive (correction to be added to the nominal volume to obtain the true volume). The huge bulk of the burets show over-all corrections of +0.05 to +0.10 ml. with a few showing corrections of +0.15 ml. or a little more. I have checked a fair number myself, especially if a student has obtained a negative correction. I have never been able to substantiate such a result. Never in my memory have I found an Exax buret with a *negative* correction. This result surprises me considerably since normal manufacturing error should be distributed more or less symmetrically about the true value.

Incidentally, the calibrations were made in the usual way by weighing the delivered water. Since room temperature usually runs between 20° and 25°C. and the water used usually is a degree or so below room temperature, a fixed factor, 1.003, was used to convert the observed weights to true volumes. This considers both the absolute density of water and an air buoyancy correction.

The above results have been communicated to the Kimble Glass Company and they have kindly expressed interest and an intention to check at the manufacturing end.

D. F. SWINEHART

UNIVERSITY OF OREGON
EUGENE, OREGON

To the Editor:

In the September, 1951, issue of the JOURNAL OF CHEMICAL EDUCATION, Mr. Milton G. Wolf of James Madison High School, Brooklyn, New York, wrote a letter to the Editor in which he made known that upon attempting to dissolve selenium in olive oil, aided by heat, he obtained later a solid material. He also stated that he was uncertain about the composition of this solid, and would welcome comment.

It seems quite possible that what he has obtained

[1] MATHEWS, FREDERICK, J. CHEM. EDUC., **28**, 258 (1951).
[2] BUTLAND, M. S., *J. New Zealand Inst. Chem.*, **13**, 53 (1949).

is the *trans* state of the oleic triglyceride instead of the normal predominating *cis* state. Oleic acid is the principal fatty acid in olive oil. It is well known that treated under proper conditions with certain oxides of nitrogen, oleic acid (either as a free acid or in the esterified state) will transform itself from the *cis* isomer (which is liquid at room temperature) to the *trans* isomer which is a solid. It may be that, at higher temperatures, selenium may also catalyze this transformation.

It seems again possible that the selenium may have combined chemically at the double bond in permanent fashion (thus destroying the double bond as such) and thereby creating something structurally akin to stearic acid, or a cross-link arrangement leading to a polymer structure.

In view of the fact that Mr. Wolf states that the reaction was carried out at high temperature (probably about 600°–700°F.) *each* of the above alternatives may have occurred, together with other possibilities such as air oxidation, etc., all of which would be likely to lead to solid endproducts from oleic esters.

JOHN P. FAIRFAX
COLLOIDAL PRODUCTS CORPORATION
SAN FRANCISCO, CALIFORNIA

To the Editor:

West (J. CHEM. EDUC., **28**, 437 (Aug., 1951)) states, in connection with the use of molecular weights in teaching quantitative analysis, that ". . . this is one of the few places were it seems safe to assume prior knowledge."

The experience of the present writer is entirely different. I have found that students come into quantitative analysis with very hazy, incomplete, or even decidedly incorrect concepts of molecular weight. One of the most common misconceptions is the mistaken idea that molecular weights can be obtained by adding the atomic weights indicated by a chemical formula—*any kind* of chemical formula. On the contrary, this method can be used only if the formula is a *molecular* formula. Adding the atomic weights represented by an *empirical* formula does not give a molecular weight. The only formula known for any salt or other electrovalent compound is the *empirical* formula, and molecular formulas are not known for such compounds. It is impossible to measure the molecular weight or to calculate the molecular formula of a salt or other electrovalent compound by any means whatsoever, because such compounds consist of *ions*, not molecules. Indeed, the concepts of "molecule" and "molecular weight" of electrovalent compounds is completely meaningless. No rational definition of either molecule or molecular weight can possibly have any significance when applied to electrovalent compounds.

The illogical and incorrect concept of "molecular weight of salts" is held by so many students in quantitative analysis that I am led to the suspicion that it is not entirely the students' fault. It is quite possible that they have learned these illogical ideas from some of the commonly used textbooks.

J. C. HACKNEY
INDIANA UNIVERSITY
EAST CHICAGO, INDIANA

BASIC ORGANIC CHEMISTRY

I. Rae Schwenck and *Raymond M. Martin*, Sacramento Junior College, Sacramento, California. The Blakiston Co., Philadelphia, 1951. ix + 323 pp. 44 figs. 14 tables. 16 × 23.5 cm. $4.50.

A NEW book in elementary organic chemistry must certainly be justified in this day. For the teacher can select almost any type of text from the large number presently available, and most likely he will find one that neatly fits his idea of how the subject matter should be presented. When an instructor believes that this is not the case, or if he feels he is in a unique situation, he inevitably becomes an author. Although Schwenck and Martin do not offer a new organization—their order of topics is conventional—they do write with the junior college student in mind. This reviewer with many years of experience in the field of junior college education feels that these students do need texts more clearly written but not less meaty. To this end "Basic Organic Chemistry" can be justified. Yet many students in all types of universities and colleges, as the preface states, are frustrated by the newness of the field and their inability to correlate principles of general and organic chemistry; above all they are disconcerted by the "Babel-like confusion of language arising from the indiscriminate use of different systems of nomenclature."

"Basic Organic Chemistry" is written for a one-semester course; ". . . it is not a 'boiled-down' edition of a text prepared for chemistry majors." In fact it caters to the periphery fields of home economics, engineering, medicine, etc. Practically all handbook data are omitted. Each chapter has its "Chapter prologue," major discussion, and then where applicable a "Reaction summary," a chart covering about half a page.

Six types of formulas are given in the first chapter on carbon. This is followed by chapters on alkanes, alkenes and alkynes,

haloalkanes, alcohols, etc. In the eleventh chapter, the carbohydrates, a very useful feature is noted, every mono- and disaccharide has both the straight-chain and ring formula side by side. Now the authors feel the student is ready for nomenclature, which is the title of chapter twelve. Aromatic chemistry is covered in the next four chapters. The book concludes with: "Heterocyclic compounds," "Industrial developments in organic chemistry," "Biological developments in organic chemistry," and "Organic reactions in vital processes." What is in reality a twenty-first chapter, for it follows the format of previous ones but is included in the appendix, is "Warfare developments in organic chemistry." The index completes the book.

It is of interest to note that and are used in the first part of the book, but in aromatic compounds and appear. On page 85 the arrow is used to show the fact that hydrogens attached to carbons alpha to a carbonyl group are more labile than other hydrogens. The same representation is found in other books; however, it seems best to confine the use of the arrow to signify coordinate covalent bonds. Some other points noted for improvement again apply to other texts as well. Symbols for carbon should be more pronounced than those for hydrogen and the valence lines should be shorter. This will enable students to make visual pictures of isomers more easily. Wurtz reactions can be used to make uneven numbered alkanes; and most important of all, treatment of a primary amine with nitrous acid is the poorest possible method for preparing primary alcohols.

The authors have fulfilled their objectives rather well and have given the students and teachers a book that is readable, teachable, and can be nicely completed in one semester. A good balance is kept between aliphatic and aromatic chemistry as well as between industrial and biological applications. Teachers who have classes predominantly of nonchemistry majors would do well to give this book consideration.

ARTHUR FURST

San Francisco City College
San Francisco, California

● SCIENCE FRENCH COURSE

C. W. Paget Moffatt. **Revised by *Noel Corcoran*. Fourth edition. University Tutorial Press, Ltd., London; Chemical Publishing Co., Inc., New York, 1951. viii + 332 pp. 12.5 × 19 cm. $4.75.**

That this is a good text of its kind is attested by its passage into a fourth edition. The elements of French grammar, employing some scientific terms in the examples, occupy 91 pages. Then follow 24 brief nontechnical essays on miscellaneous topics ranging from the seasons to fishing in Iceland. Of the selections from journals and B.Sc. papers physics gets 56 pages, chemistry 32, and some other sciences smaller space.

Apparently about the only change in this edition is the adding of some 150 words to the vocabulary, which, however, is still incomplete. Some of the terms noted in the chemical section but not in the vocabulary are benzénique, brome, bromuration, cyanure, diazoïque, eaux-méres, étalonner, filtrer, groupement, huileux, iodé, iodure, revêtement. (One must admit, by way of excuse, that a complete vocabulary in a foreign-language reader is an exception.) The survey of grammar is well handled and the printing is attractive.

One may ask, of what value is such a course? Its announced purpose is "to provide students who have to read French books on scientific subjects with the necessary minimum of grammar and a selection of extracts from which a little preliminary practice may be obtained." It does just that, but the claim on the jacket, "This book enables students without any previous knowledge of the language to read French scientific and technical literature," is not justified. The reviewer's experience with "science readers" for science majors has been disappointing. This one may provide pleasant reading for a student who wishes practice in French and who is interested in science, but it will give him only a smattering of scientific terms. The difference between ordinary and scientific French lies in the technical vocabulary of the latter. As a rule, a science student who has French articles or books to read has a main interest in a particular branch of science. For him, in the writer's opinion, a "science French course" would be largely a waste of time. He had better secure a foundation in general French and then get down to the French of his specialty. In this way he will be improving his general knowledge of the language and at the same time acquiring an adequate technical vocabulary in a particular subject. This vocabulary can of course be extended to other scientific subjects as occasion warrants. The same considerations apply to the study, as a tool subject, of German or any other foreign language.

AUSTIN M. PATTERSON

Antioch College
Yellow Springs, Ohio

● PLASTICS, SCIENTIFIC AND TECHNOLOGICAL

H. R. Fleck. **Third edition. Temple Press, Ltd., London, 1951. xv + 414 pp. 76 figs. 14.5 × 22.5 cm. 40s.**

The new edition of this standard text is a welcome addition to our growing literature on plastics. It continues to be a balanced summary of plastics information up to 1949, with special reference to the British position.

After a very cursory survey of raw materials and theoretical principles, the book treats the chemistry of plastic materials in more detail. Laboratory procedures are given. Fourteen pages are devoted to the manufacture of plastics, and 69 pages to physical properties. Chapters follow on "Synthetic resins," "Synthetic fibers and textiles" (which is particularly inadequate and obsolete), "Adhesives and plywood," "Moulding techniques," and "Chemical analysis and testing."

Very little material in this book cannot be found in other more detailed, and more modern, references. The primary attraction of this book is that it is a bound compilation which can serve as an "additional" reference. The U. S. book literature has been so abundant since the previous editions of this volume that it has lost much of its attractiveness and value. It is recommended to libraries and technical men who should have all the books on plastics published here and abroad.

ROBERT S. ARIES

Polytechnic Institute
Brooklyn, New York

● HETEROCYCLIC COMPOUNDS. VOLUME I: Three-, Four-, Five-, and Six-Membered Monocyclic Compounds Containing One O, N, and S Atom

Edited by *Robert C. Elderfield,* Columbia University. John Wiley & Sons, Inc., New York, 1950. vii + 703 pp. 4 figs. 1 table. 4 charts. 15.5 × 24 cm. $11.

This is the first volume of a proposed series concerned with the chemistry of heterocyclic compounds. The editor of this series has recognized and has stated in his preface that ". . . a definite need existed for a detailed treatment of the chemistry of these interesting compounds which would concentrate on the chemical principles involved rather than attempt to give an encyclopedic coverage of the field."

Apparently, the present volume begins an attempt to fulfill this stated need by providing a series of critical reviews, each of which is to deal with one or more heterocyclic ring systems. This first volume contains separate discussions of the following topics: Ethylene and Trimethylene Oxides (60 pages), Ethyl-

eneimine (17 pages), Derivatives of Azete (41 pages), Furan (89 pages), The Chemistry of Thiophene (69 pages), The Chemistry of Pyrrole and Its Derivatives (66 pages), Monocyclic Pyrans, Pyrones, Thiapyrans, and Thiapyrones (54 pages), The Chemistry of the Pyridines (220 pages), and Piperidines and Partially Hydrogenated Pyridines (60 pages). Thus all of the ring systems indicated in the title except the three- and four-membered rings containing one sulfur hetero atom have been covered. This omission is justified because of the very small amount of published information concerned with these heterocyclic sulfur compounds. The number of pages allotted to each of the various other topics probably represents an attempt to rank the importance of the various heterocyclic systems according to amount of published information available and according to present research interest. For example, 88 references to books and journal articles are listed in the 41-page chapter concerned with the derivatives of azete, while 285 references to similar sources are given in the 54-page chapter entitled "Monocyclic pyrans, pyrones, thiapyrans, and thiapyrones." Derivatives of azete have become important recently because of the discovery that a β-lactam ring is present as part of the fused ring system of penicillins. For this reason and in spite of the actual number of publications concerned, derivatives of azete are worthy of the relative number of pages permitted them in this volume. The scope of the chapter concerned with azete and its derivatives, however, does not include actual discussion of penicillins or other compounds with azetidine rings as part of a fused ring system.

Although the two chapters concerned with pyridines, piperidines, and partially hydrogenated pyridines have been written by the same author, each of the other chapters has been written by a different author or pair of authors. There has been little attempt to relate the material of one chapter with that of the others. The reader will seek in vain for adequate comparisons of the heterocyclic systems. For example, any detailed treatment of heterocyclic compounds which proposes to "concentrate on the chemical principles involved" might well include a discussion of the dependence of chemical reactivity of heterocyclic compounds on the nature of the hetero atom, whether oxygen, sulfur, or nitrogen.

The multiple authorship has also caused some irregularities in the scope of each chapter. For example, the chapter concerned with azete and its derivatives contains extensive material on β-lactams, while the chapter on ethylene and trimethylene oxide omits discussion of β-lactones. Although biotin and related work with tetrahydrothiophenes is allotted a total of 27 pages in the chapter entitled "The chemistry of thiophene," only slight attention is given to the important reduced furans and to pyrrolidines in the chapters concerned with these ring systems. Further, the molecules of most alkaloids and many other natural products have heterocyclic rings. With the exception of the extensive discussion of the structure elucidation and syntheses of biotin, such natural products are discussed only when they serve to illustrate important points of heterocyclic chemistry.

In spite of the fact that it is not the aim of this volume to provide exhaustive bibliographies concerning the various heterocyclic ring systems, it is obvious that the chemical literature has been carefully covered and reference made to the important sources of chemical information. The pertinent patent literature seems to have been covered. For example, of the 800 literature references in the chapter entitled "The chemistry of the pyridines," 67 direct attention to patents.

A useful, well-organized, 27-page index has been included. This is largely an index of compounds and the nomeclature used is that discussed at the beginning of each chapter. With a few exceptions, this nomenclature follows the recommendations of *Chemical Abstracts*. It may be noted here, however, that the time has come to stop using such names as pyromucic acid in place of the simpler and more meaningful 2-furoic acid merely because they have been used in the older chemical literature. On the other hand, dehydroacetic acid may still be preferred to 3-acetyl-6-methyl-1,2-pyran-2,4(3H)-dione. The fine quality of the binding and paper as well as the excellence of the typography, especially in the presentation of structural formulas, is in keeping with the publisher's usual high standards in such matters There are only a few typographical errors. This reviewer does not agree with the simple explanation given on page 152 for the peculiar orientation observed during various alkylation reactions of furfural. This is clearly an example of a "wild flight of theoretical speculation" which the editor has promised in his preface to avoid.

On the whole, this book is an excellent contribution to the chemistry of organic heterocyclic compounds. It will be very useful as a textbook for students in the increasing number of graduate chemistry courses concerned entirely or partly with organic heterocyclic compounds. Use of this book will also mean a great saving in time to teachers of such courses. It may be expected with confidence that this series of critical reviews will stimulate considerable research in an already rapidly expanding branch of organic chemistry. Chemists actively engaged in research with one heterocyclic system will probably benefit greatly by studying the discussions of the other systems. It seems worthy of note, however, that considerable duplication of effort may result by continued publication of this series and a similar series entitled "The Chemistry of Heterocyclic Compounds," now appearing from Interscience Publishers with A. Weissberger as editor.

EARL C. SPAETH

UNIVERSITY OF CONNECTICUT
STORRS, CONNECTICUT

● ESSENTIALS OF CHEMISTRY

***Alfred B. Garrett, Joseph F. Haskins,* and *Harry H. Sisler,* Department of Chemistry, The Ohio State University. Ginn and Co., Inc., Boston, 1951. vi + 569 pp. Illustrated. 16.5 × 24 cm. $5.**

THIS book is aimed at answering the question: "What should a student gain from the study of science that will make him a happier, better-informed citizen of his community?" It is intended for use in a beginning course which is also a terminal course in chemistry.

The contents are organized into eight units: "The materials of which the world is made," "The components of the atmosphere," "The states of matter," "The material of the earth's crust," "Radioactivity and atomic structure," "The periodic system: the families of the elements," "Organic chemistry: the chemistry of the compounds of carbon," and "The chemistry of everyday life."

Much of the usual material on writing chemical equations is included in an extensive appendix together with a discussion of arithmetical methods. This is consistent with the general tenor of the book which avoids a heavy accent on problem working, yet the more fundamental problems of molecular weight determination, stoichiometry, and volume relationships are all introduced so that each student using the book will be well acquainted with the necessity of exact quantitative work in the physical sciences. However, the general accent on qualitative comprehension of principles avoids the common error of attempting to make all beginning chemistry students into proficient calculators.

Most of the material is presented by stating the applicable theory and then showing how the facts support it. The authors have done an excellent job of selecting for discussion facts and processes which the student already knows in a general way or which he will undoubtedly meet in the present world, and the economic and social implications of advances in chemistry are effectively illustrated. To each chapter is appended a description of pertinent film strips and movies, a supplementary reading list, and a set of problems.

Potential users of the text will probably be strongly swayed in their judgment by the relative accent on inorganic and organic chemistry. Such old standbys of inorganic chemistry as the Solvay process, the Lead Chamber process and the Contact process rate only 30 lines all together, which will shock some teachers, particularly when they note that over one-quarter of the book is

devoted to organic chemistry. Yet this redistribution of space is certainly consistent with so-called "everyday chemistry" in the present world.

Liberal use of bleed printing of pictures, wash drawings, and sketches of atomic and molecular models gives an exceptionally pleasing format. Not only is the eye-appeal increased, the figures are well chosen and, with few exceptions, replace a great many words. A clear style of writing, well chosen vocabulary, and narrative continuity combine to give a fruitful source of knowledge for those whose training in chemistry will end here. It is too much to hope that each student using this book will, as a result, be happier, but he should be better informed.

J. A. CAMPBELL

OBERLIN COLLEGE
OBERLIN, OHIO

● ADVANCED ORGANIC CHEMISTRY

Reynold C. Fuson, **Professor of Organic Chemistry, University of Illinois. John Wiley & Sons, Inc., New York, 1950. x + 669 pp. 16 × 24 cm. $8.**

IN THE author's words, "This book is designed for the use of students who have had an introduction to organic chemistry and seek a mastery of the fundamentals of the subject. The author believes that the primary problem of the advanced student is not the acquisition of extensive knowledge of the many specialized fields of organic chemistry but rather the intimate aquaintance with facts and theories which is essential to success in research. Accordingly he has sought to bring together those features of the subject that constitute the least common denominator of the permanent equipment of investigators in the field."

The treatment is well adapted to the needs of advanced undergraduate and beginning graduate students, being neither encyclopedic nor merely a slight expansion of an elementary text. Aliphatic and aromatic chemistry are intermingled with a satisfactory balance between the examples taken from each branch.

The gross organization of the book is not conventional, for it is the author's opinion that students will learn more by reexamining the facts in an unfamiliar context, and this is probably true. The chapter titles are: "1. Introduction," "2. Cleavage of carbon-carbon bonds," "3. Aliphatic substitution," "4. Carbon-carbon multiple bonds," "5. Hydroxy compounds," "6. Halogen compounds," "7. Organometallic compounds in synthesis," "8. The ether linkage," "9. Carbon monoxide in synthesis," "10. Derivatives of carboxylic acids," "11. Oxidation," "12. Hydrogenation, dehydrogenation, and hydrogenolysis," "13. Substitution in the aromatic series," "14. Alkylation of aromatic compounds," "15. Acylation of aromatic compounds," "16. Carbonyl compounds," "17. Ring closures involving condensation of carbonyl groups with aromatic nuclei," "18. Active methylene compounds," "19. Conjugate addition," "20. Nitro, nitroso, and oximino compounds," "21. Amines and amino compounds," "22. Azo and diazo compounds," "23. Organic sulfur compounds," "24. Aromatic character," and "25. Polymerization." Although the selection of topics is excellent, the implication that they "constitute the least common denominator of the permanent equipment of investigators in the field" should not be taken too seriously.

The specific examples discussed under each topic are, perhaps, less carefully chosen, although they do serve the purpose reasonably well. Largely, they are items either that have been in recent journal articles or that are in "Organic Syntheses," which obviously was combed in detail, for the text abounds with examples taken from it.

The treatment is definitely more descriptive than theoretical. Both the statement from the preface quoted above and the promotional material issued by the publisher seem to give the impression that the book contains more of the theory of organic chemistry than it does. The publishers would do better to advise all serious advanced students to buy also their other "Advanced Organic Chemistry" by Wheland. A careful study of the two books would give such students a fairly well-balanced idea of the present status of organic chemistry, and would even put them on the road toward acquiring that "intimate acquaintance with facts and theories which is essential to success in research." The theoretical material that is interspersed in the present volume will often only puzzle the students for whom this book is intended, since the usual first course in organic chemistry will not have given them enough background to understand it without further elaboration.

The nomeclature of organic compounds is completely neglected, in spite of the obvious fact that a knowledge of systematic nomenclature is a very important, although possibly small, part of the "least common denominator of the permanent equipment of investigators in the field."

The book is moderately well documented, averaging somewhat more than one footnote reference per page. In addition there are at least an equal number of references to "Organic Syntheses" and a few to the three other sources that are cited in the body of the text rather than in footnotes.

The coherence leaves something to be desired, the relationship between successive paragraphs and the like not always being clear. In fact, there is sometimes the impression of a series of more or less isolated observations written down at different times and then arranged in some logical order without much rewriting or introduction of connectives. Even the experienced chemist will sometimes be bewildered temporarily over the relationship of a paragraph or statement to what has just preceded it and to the unit as a whole. It is quite possible that this bewilderment may for the student be more than temporary.

In spite of such minor defects, the text is an excellent one—one of the best yet written on the advanced level. It is well printed and is remarkably free from typographical errors. It seems also to attain a high standard of factual accuracy.

LAWRENCE H. AMUNDSEN

UNIVERSITY OF CONNECTICUT
STORRS, CONNECTICUT

● INTRODUCTORY CHEMISTRY

Lillian H. Meyer, **Western College of Michigan, Kalamazoo, Michigan. The Macmillan Co., New York, 1951. x + 532 pp. Illustrated. 14.5 × 22 cm. $5.**

THIS text fills a need which has existed for some years. It is about a decade since even a revised edition of an elementary chemistry book for home economics students has appeared. The author suggests that it may be used also with other groups interested in the applied biological sciences. This suggestion is pertinent to college students in nursing and physical education programs. Dr. Meyer uses the text for a year's course in chemistry, which is the maximum amount usually permitted such students. In fact, she does not find time to include all work given therein, but has added some material so that other instructors can make a selection, according to her preface.

The content of the book is wisely chosen. Over half of the text is included under the heading of "Introductory Chemistry." The balance consists of approximately 10 per cent organic chemistry, 20 per cent biochemistry, and 6 per cent textile chemistry. The index and a short appendix complete the book. The Introductory Chemistry section is subdivided into 21 chapters. Each of these treats a topic important in the subsequent work of the students such as solutions, the colloidal state, leavening agents, etc., instead of attempting to survey the whole general and inorganic chemistry field. In other words, the author has assisted the teacher in the selection of appropriate subject matter. This choice becomes increasingly difficult as the body of chemical knowledge grows each year. Further help is given the teacher by occasional comments as to the extent to which certain reactions may be used in our country. For example, electrolysis of water is of little industrial importance because of the relatively high cost of electric power. The author has excluded certain topics which are not of great value to the students for whom the

text is written. The methods of determining atomic weights is a case in point. However, there is sufficient use of mathematics and its methods to give the students an appreciation of its importance in chemistry and practice in its use. The review questions at the end of each chapter contain some problems. Incidentally, the practical nature of these questions should appeal to the instructor. A list of "New Terms" which accompanies these questions should be valuable to both teacher and student.

The relatively brief organic chemistry section is sufficient to introduce the student to the more important biochemistry division. The chemistry of the major compounds of foods (proteins, carbohydrates, and fats) and their fate in digestion, metabolism, and excretion is well and concisely covered. The textile portion discusses various kinds of fibers and presents a brief treatment of dyeing, laundering, and cleaning textiles. This material should interest all students in this day of synthetic as well as natural fabrics.

In conclusion, the author has done a splendid piece of work which should meet with wide approval from the group for which the book has been written. The conciseness and clarity of its style alone should win it favor. Its applications are excellent.

HELEN I. MINER

WAYNE UNIVERSITY
DETROIT, MICHIGAN

• RADIOISOTOPES: INDUSTRIAL APPLICATIONS

G. H. Guest, Senior Scientific Officer, Health Radiation Section, Industrial Health Division, Dept. of National Health and Welfare, Ottawa, Canada. Pitman Publishing Corp., Toronto and New York, 1951. xv + 185 pp. 65 figs. 9 tables. 15 × 22.5 cm. $4.50.

"WITH the development of the cyclotron and the construction of atomic piles, scientists have created nearly six hundred new isotopes of the ninety-two naturally occurring elements. . . . These radioactive atoms have provided a new tool, millions of times more sensitive than conventional chemical and spectroscopic methods, with which scientists, engineers, and technicians may follow reactions and investigate phenomena revealing new facts and important applications. In this book, Dr. Guest has given in simple language the basic ideas involved in the use of these new isotopes, indicating typical examples of their application in industry. . . ." Thus writes Dr. D. A. Keys, Vice-President of the National Research Council of Canada, and Director of the Atomic Energy Project, Chalk River, Ontario. As the author himself puts it, "industrial applications of radioisotopes are already varied and widespread and the purpose of this book is to stimulate further interest in uses of pile-produced isotopes in industry."

Written in language which an intelligent layman can comprehend, the book is slanted toward the industrialist looking for possible applications of the radioisotope technique. The book is equally well suited to the professor giving a course in radiochemistry; many experiments are suggested which in addition to supplying valuable information both of a fundamental and applied nature, would serve also to illustrate the use of tracer techniques. The book not only provides a fairly complete summary of experiments already suggested or tried but the author goes on to suggest many which could be tried. As a general survey of the field, the book is excellent. It is not meant to be a comprehensive text discussing in detail counting techniques, etc.; neither does the author go into detail about the results of the experiments which he describes, but rather uses them only as illustrations of the tracer method. In the opinion of the reviewer it would have been desirable to go into a bit more detail on the last two chapters entitled "Laboratories." and "Precautions in the industrial uses of radioisotopes."

The format is good. The clear type together with a lavish use of well chosen figures combine to make a very readable book. Numerous references are given at the end of each chapter and in the appendix. Although the index is somewhat weak, it is offset by a very complete table of contents. Only a few minor points of contention were noted: (1) the reviewer does not feel that painted concrete floors are adequate for a radioisotope laboratory as is suggested on page 142; (2) on page 33, the author neglects to mention consideration of the type of radiation (α, β, or γ) in deciding whether an isotope would be of use in a metallurgical problem. The reviewer commends the use of the term "autoradiography" rather than the "radioautography" that is used in this country.

In general the book is a useful one and is recommended.

LOUIS G. STANG, JR.

BROOKHAVEN NATIONAL LABORATORY
UPTON, LONG ISLAND, NEW YORK

• QUANTITATIVE ORGANIC MICROANALYSIS

Based on the methods of *Fritz Pregl*. Fifth English edition. Revised and edited by *Julius Grant*. The Blakiston Co., Philadelphia, 1951. ix + 342 pp. 183 figs. 15.5 × 24 cm. $5.50.

THE fifth English edition of this classic work is appreciably enlarged but similar in scope to the fourth edition (reviewed in J. CHEM. EDUC., **23**, 468 (1946)). It incorporates advances in the subject made in certain European countries during the war years and more recently released as well as the most recent literature. Several determinations are cited here for the first time, notably the direct determination of oxygen by its conversion to carbon monoxide which reacts with iodine pentoxide to free iodine.

Especially to be noted is the inclusion of the full report of the Committee for the Standardization of Microchemical Apparatus of the American Chemical Society as well as a detailed discussion of British standards prepared by the British Standards Institution. These reports include drawings with full specifications of microchemical apparatus.

The inclusion of physical methods adds to the value of a book of this kind. While the discussion in this chapter is intentionally brief, the reviewer feels that in another edition the section on absorption spectra should be revised as there is inaccuracy of statement in the discussion of the Beer-Lambert law.

The text is primarily a treatment of microanalysis from the viewpoint of organic chemistry and as such plays an important role in the development of this subject.

LUCY W. PICKETT

MOUNT HOLYOKE COLLEGE
SOUTH HADLEY, MASSACHUSETTS

• THE POWER AND LIMITS OF SCIENCE

E. F. Caldin, Lecturer in Chemistry in the University of Leeds. Chapman and Hall Ltd., London, 1949. ix + 196 pp. 14 × 22 cm. 12s. 6d.

THIS book, subtitled "A Philosophical Study," is timely, well written, and stimulating. Probably few scientists would agree with all of the author's premises, arguments, and conclusions but few could fail to profit from a critical and careful reading of the book.

Its values are of two distinct types: the ideas discussed and the clarity of the writing itself. The ideas should help to stimulate and crystallize thoughts of the reader, although he will not always agree with the author. The writing itself could be studied by many as a model.

Some of the flavor of the book can perhaps be communicated—although only imperfectly—by quoting excerpts such as the following, from a chapter entitled "The inductive method." "The view that we are developing is that scientific generalisations are neither certainly false nor certainly true, but more or less reliable or likely, according to the evidence supporting them; that a scientific law claims a qualified belief, and the likelihood of its being true can be improved or worsened by fresh evidence. All such likelihood depends, too, not only on the bare data of science, but upon the legitimacy of assuming a principle which

may be expressed in some such form as 'there is order in nature.' This is not, indeed, . . . a *sufficient* condition for a generalisation to be *certain;* but it is a necessary condition without which no empirical generalisation would have any likelihood—without which science could not begin" (pages 59–60).

". . .This belief that there is order in nature is not a conclusion, but a presupposition of science; induction cannot begin without it. Yet neither formal logic nor the bare observations warrant it" (page 61). ". . .The grounds for believing this presupposition must be sought in metaphysics. . ." (page 70). (This last statement is that one of the author's fundamental contentions with which many scientists are most likely to disagree.)

Many of the summaries are gems, models which could well be studied by many authors. To judge a summary without reading the discussion it follows is of course not fair. Nevertheless, here is the summary of the 25-page chapter, "Society and science": "Science is distinct from technology, and the social problems raised by it are quite different. The social function of science is twofold: to represent rational values and to supply techniques for legitimate material ends. Science is concerned in the major crisis of our time—the decline in our conception of man—because the value of truth is endangered, and because the crisis is partly due to the belief that science is the sole means of reaching truth, so that a more accurate view of science would remove one source of low views of the nature of man. Science can help to a solution of the crisis—the recovery of a true view of man—in so far as it favours respect for truth and for the human person. Scientists should make it their business to uphold rational values, and at the same time resist attempts to extend scientific method to fields where it is inappropriate and can only lead to disastrous conclusions. Science should find a place in a liberal education, which is incomplete without a view of nature; the deficiencies of its method can be supplied by teaching it in association with other disciplines" (page 174).

Most scientists would probably agree for the most part with the author's analysis of science, but many would feel that the book should be accompanied by a companion volume: "The Power and Limits of Philosophy." Nevertheless, "The Power and Limits of Science" is well worth reading.

WILLIAM E. MORRELL

UNIVERSITY OF ILLINOIS
URBANA, ILLINOIS

● ORGANIC REACTIONS. VOLUME VI

***Roger Adams,* Editor-in-Chief. John Wiley & Sons, Inc., New York, 1951. viii + 517 pp. 5 figs. 72 tables. 15.5 × 24 cm. $8.**

FIVE previous volumes in this series have familiarized chemists with the style and scope to be expected in each new volume. This volume lives up to the previous high standards.

The first chapter deals with the base-catalyzed condensation of aldehydes (ketones) with succinic esters, or the Stobbe condensation. The chapter is written by W. S. Johnson and G. H. Doub. It is exceptionally well done. Coverage is complete, presentation well organized and interesting, and nomenclature is sound throughout.

This chapter is followed by two chapters by W. M. Whaley and T. R. Govindachari on dihydroisoquinolines and tetrahydroisoquinolines and one by W. J. Gensler on isoquinolines. The 132 pages devoted to various syntheses of isoquinolines and related compounds reminds one of the little girl's report on a book dealing with penguins. She said simply: "There is more about penguins in this book than I care to know." Of course, penguin fanciers might wish for 200 pages rather than 132.

Carl Djerassi's chapter on the Oppenauer oxidation brings out the fact that it has been used in the past primarily for the oxidation of secondary alcohols to ketones in the steroid series. Other chapters include syntheses of phosphonic and phosphinic acids (G. M. Kosolapoff), halogen-metal interconversion reaction with organolithium compounds (R. G. Jones and H. Gilman), thiazoles (R. H. Wiley, D. C. England, and L. C. Behr), thiophenes and tetrahydrothiophenes (D. E. Wolf and K. Folkers). The volume closes with an authoritative treatment of reductions by lithium aluminum hydride by Weldon G. Brown.

Each contributor mentions the date through which the literature has been reviewed. Six chapters include 1949 references, two include 1948 ones, but two (thiazoles, thiophenes) unfortunately are complete only through 1946. This results in one serious omission in the thiophene chapter, namely, the noninclusion of the process developed by The Texas Company for the synthesis of thiophene.

Again, the editors are to be congratulated for having sponsored so useful a book, and the publishers for preparing it so attractively.

CHARLES D. HURD

NORTHWESTERN UNIVERSITY
EVANSTON, ILLINOIS

● ECONOMIC ASPECTS OF ATOMIC POWER

***Sam H. Schurr* and *Jacob Marschak.* Princeton University Press, Princeton, New Jersey, 1950. xxvi + 289 pp. 32 tables. 4 maps. 2 graphs. 17.5 × 26.5 cm. $6.**

IN THIS book the authors present the results of a study which they directed for the Cowles Commission for Research and Economics. Three research associates (Sam H. Schurr, George Perazich, and Edward Boorstein) and three research consultants (Herbert A. Simon, Harold H. Wein, and Milton F. Searl) are listed as contributors to the study. In addition, an extensive list of the sources of information, persons, and organizations is given in the preface. The presentation of the study is straightforward, matter of fact, and gives a dispassionate view of the probably small total effect of atomic power on the world's economy, a relief from the bulk of literature on atomic energy.

The core of the study is the estimate in Part I of the cost of power from nuclear fuels. The estimate is introduced by a consideration of the characteristics and uses of atomic power, the nature of plant and equipment required, and the supply of raw fuel. It is established that the minimum cost will be that of conventional power minus fuel costs, but that fixed charges will make the minimum difficult to realize, at least without a long development period. Thus a minimum cost of atomic power in the form of electricity would be 4 to 4.3 mils per kw.-hr. in 1946 prices. With slight changes as to assumptions allowing for the effect of higher fixed costs, the price range is 6.6 to 7 mils per kw.-hr. which may be given as an intermediate cost. A maximum cost is given as 10.2 mils per kw.-hr. and takes into account the possible high cost of processing fuel. The authors cope very fairly with the lack of definitive information on which to base these estimates. They point out this lack is due more to the fact that developments have not taken place as yet rather than due to security regulations. The technical man reading the book will find himself desiring specific reactor designs to which to relate the discussion. The interaction between weapons production and power production is not discussed. Following the discussion of the cost of atomic power, the cost of conventional power in various parts of the world is analyzed thoroughly and presented in instructive fashion.

In Part II the effect of cheaper power (electricity and heat), together with the effect of its redistribution on selected industries, is evaluated as follows:

Aluminum. The future expansion of aluminum output in the United States might be supported by the availability of large amounts of atomic power. Should the expansion take place due to this fact, it would be in locations differing from those being considered today.

Chlorine and Caustic Soda. At most a 5 per cent reduction in cost may be expected in a few areas, hence little or no over-all effect is predicted.

Phosphate Fertilizer. No significant effect is anticipated although some change in methods of production may be possible.

Cement. Except in case of coincidence in the location of a nuclear reactor and the cement mill no effect is anticipated.

Brick. Little or no effect since brick plants are small.

Flat Glass. Use of atomic power would probably be disadvantageous although a very small reduction in cost might be possible.

Iron and Steel. By possibly combining the development of the sponge iron process with the development of electrical power for the nuclear reactor to produce hydrogen, an effect on the location of steel production and the utilization of iron ore is foreseen as a possibility. No other effect is anticipated.

Residential Heating. In some areas nuclear heating might provide convenience but in any event little or no reduction in cost.

Of more value than learning the effects of atomic power on the selected industries is the analysis of the importance of power, its location and distribution on each industry. The methods developed by the authors should be of considerable utility to others.

Part III attempts to see how our regional and national economy might change were atomic power to become widely used. Results of Part I and II are used and factors including effects which might trigger new developments are estimated. Outside of trigger effects there will be no short-term effects. Longer-term effects become very speculative and depend heavily on the stimulation of invention leading to the introduction of new power-consuming devices. Some increase in income is expected, however, in the long term, and if this also results in a more rapid accumulation of capital, effects of fairly large magnitude might be expected in our economy. However, the cheapening of power alone will not produce these effects.

The book is scholarly and written primarily from the point of view of the economist. A tremendous volume of data has been gathered, analyzed, and presented in orderly fashion. To the technical man who thinks of new developments as important not so much because they save money but rather because they enable us to do something previously impossible and probably not even considered previously, the book is somewhat discouraging. As mentioned by the authors, it is the stimulation of inventions and the interaction of new developments that makes them important. It is likely that the development of atomic power will come about because of its special applications rather than because of general effects on our economy. Thus the reviewer feels that the information, the balanced point of view, and in particular the techniques for handling the analyses of the effects on national or regional economies are far more important than the conclusions drawn if they were solely to be considered.

JOHN P. HOWE

General Electric Company
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• AN INTRODUCTION TO TEXTILE FINISHING

I. T. Marsh, Manchester, England. John Wiley & Sons, Inc., New York, 1951. xv + 552 pp. 161 figs. 14 × 22.5 cm. 27 plates. $5.50.

The present text is a companion volume to "An Introduction to Textile Bleaching" written by the same well-known British textile authority. As stated in the preface "the subject matter of this book is based on the experiences of the writer during his twenty years' service as a research chemist in the textile industry, and from a careful study of the literature of the subject." "Two criteria have been taken for this work, first, commercial importance, and secondly, scientific and technical interest." The book deals principally with the chemical aspects of textile finishing although some consideration of the mechanical aspects is necessarily included.

After describing the principal types of finishing machinery, the processes of creping, mercerizing, parchmentizing, preshrinking, softening, starching, weighting, and delustering are considered. Also the setting, milling, and giving to wool an unshrinkable finish are presented. The reaction of formaldehyde with different fibers is described and also the application of cellulose derivatives, resins, and rubber to the finishing of fabrics. Then the proofing of fabrics against water, moths, mildew, and fire is considered. Included are a bibliography of finishing and name and subject indexes. The book is well illustrated with figures and plates, including one in color.

This book brings together a large amount of previously scattered material dealing with the finishing of cotton, wool, silk, linen, and rayons; however, the newer polyamide, polyester, acrylic, and vinyl fibers receive little or no attention. As is natural the coverage for the British patent literature is much more complete than for United States patents. The author's wide knowledge of textile finishing has enabled him to make a very worth-while contribution to this field and this book should find a place in every finishing plant and textile library.

W. W. RUSSELL

Brown University
Providence, Rhode Island

THE VITAMIN B COMPLEX

F. A. Robinson, Deputy Director of Research, Allen and Hanburys Ltd., England. John Wiley & Sons, Inc., New York, 1951. xi + 688 pp. 14 × 22 cm. $9.

This book presents a wealth of detailed, systematized information regarding vitamins of the B group. With true British conservatism the author uses the British pharmocopoeia name, aneurine, for what in America is called thiamine, yet he tacitly acknowledges the appropriateness of thiamine in several ways: *e. g.*, he writes of thiaminase. No formal definition of the B group of vitamins is attempted, but in general the definition as accepted by R. J. Williams seems by implication to prevail in the author's mind.

Twelve chapters, of which the first is the longest, deal respectively with aneurine, riboflavine, nicotinic acid, pyridoxine, pantothenic acid, biotin, the folic acid complex, vitamin B_{12} (erythrotin), *p*-aminobenzoic acid, inositol, and choline.

The thirteenth chapter is entitled "Miscellaneous water-soluble growth factors." It presents brief accounts of experiments leading to the coining of names that are now abandoned and of other experiments suggesting the existence of food factors that have not yet been definitely established. One might wish that the story of vitamin B_{14} and its significance had been more fully developed. The last chapter is a useful and stimulating general view of this field.

The author has done a fine job in documenting the book. Full lists of references are conveniently placed at the ends of the several sections into which each chapter is divided. Indeed, the book is of great value as a working bibliography in addition to its valuable text.

All workers in nutrition and in vitamin research will find this book extraordinarily useful as a reference treatise.

PHILIP H. MITCHELL

Brown University
Providence, Rhode Island

• THE ATOMIC-MOLECULAR THEORY

Leonard K. Nash. Harvard University Press, Cambridge, Mass., 1950. v + 115 pp. 15.5 × 23 cm. $1.35.

This is number four of a series of "Case Histories in Experimental Science." Numbers one and two were prepared by the editor of the series, James B. Conant, and appeared in his book, "On Understanding Science: An Historical Approach"; number three was on the "Early Development of the Concepts of Temperature and Heat," prepared by Duane Roller.

The editor of the series considers their purpose "to assist the student in recapturing the experience of those who. . . participated in exciting events in scientific history. . .(Thus) by the

intensive study of the actual work of. . .the past. . .the methods then employed (may) stand out clearly. . .(By such) an introduction. . .the inquiring citizen ought to be in a position to continue his scientific education throughout his life."

This booklet, after proper emphasis on the antiquity of the atomic concept, devotes its first section to Dalton's "deceptively simple proposals" which justify calling Dalton "the effective architect of the. . .theory as we know it today."

Sections two to five present the theory's critical reception by chemists of that time, Gay-Lussac's empirical whole-number volume law of reacting gases (which Dalton rejected principally because it did not correlate well with results from his "rule of greatest simplicity"), and Avogadro's hypothesis, which was also rejected by Dalton for the same reason.

In an epilogue, section six, Davy's success in the electrolysis of alkali salts and Berzelius' theory of electrical force as the binder of elements into compounds (so-called dualistic theory) are credited with having held off for 40 years the effective use of the law of reacting gases and Avogadro's hypothesis in solving Dalton's problem of consistent atomic weights. This problem was finally solved convincingly by Cannizzaro in 1858, and this accomplishment was the closing act of this bit of scientific history.

There is neither a table of contents nor an index. In the preface suggestions are made with reference to the booklet's use with classes in "general education courses in physical science."

The reviewer found the book a very helpful supplement to his own knowledge of this theory's evolution and anticipates that teachers of physical science in general will welcome its assembly of these source materials. He found the treatment a bit tedious and involved in a number of places and frequently was uncertain whether certain passages were direct quotations from the sources or were simply the author's exposition. He wonders if students will find this account correctly described as "exciting events in scientific history." Would an "inquiring citizen" have the persistence to read it through? Should not this "case" more correctly be offered to the physical science major after he has had his sequence of scientific courses?

B. CLIFFORD HENDRICKS

University of Nebraska
Lincoln, Nebraska

● LES MÉTHODES D'ANALYSE DES RÉACTIONS EN SOLUTION

G. Charlot and _R. Gauguin_, Professeur and Sous-chef de Travaux respectively, l'École Supérieure de Physique et de Chimie industrielles. Masson et Cie, Paris, 1951. viii + 328 pp. 242 figs. 1 table. 17 × 25.5 cm. 2200 fr.

The authors believe that a knowledge of the nature of the chemical species existing in a solution, and of their reactions, is important, not only for research, but also for industrial problems involving such systems, including the instrumental control of chemical processes. Consequently, this book presents the authors' summary of methods which have been used in studying many different kinds of liquid solutions for such purposes.

Chapter I lays the foundation for general reactions, such as oxidation-reduction and complexation (including protonation). Electrons, ions, and polar molecules are the reactants. This is followed in Chapter II by mathematical treatments of various types of possible reactions, including the methods of calculating equilibrium constants. The appendix contains 27 pages of such constants. This general material is completed in Chapter III, which deals with the graphical presentation of data obtainable when reactions proceed in solution. Different kinds of titrations and Job's method of continuous variations are examples.

Chapter IV considers possibilities of applying more than 25 different instrumental methods. The general principle of each is stated, followed by specific examples of recent applications, including original references.

More detailed applications follow, Chapter V covering various redox and complex systems, and Chapter VI dealing with reactions involving a precipitation. The next four chapters deal mostly with the use of different solvent systems and added indicator compounds.

Chapter XI covers information deducible from measurements of the heat of reaction of solutions, and Chapter XII closes with a consideration of irreversible reactions, especially types involving oxidation-reduction or change of ion composition.

In addition to the extensive table of equilibrium constants, the appendix summarizes the significance of activity of solution components and lists a glossary of terms used.

Unfortunately, the paper binding will not withstand much wear, and there is no index. This is one case where both author and subject indexes might well have been added.

To the reviewer this book seems the best summary available covering our knowledge of the means and methods for studying the nature and action of the entities which can exist in a solution. He has long needed such a work. The extensive references to the literature should be of value.

M. G. MELLON

Purdue University
Lafayette, Indiana

● ENCYCLOPEDIA OF CHEMICAL TECHNOLOGY. VOLUME VII: FURNACES TO IOLITE

Edited by _Raymond E. Kirk_ and _Donald F. Othmer_, Professors and Heads, Departments of Chemistry and Chemical Engineering, respectively, at the Polytechnic Institute of Brooklyn. Assistant Editors: _Janet D. Scott_ and _Anthony Standen_. The Interscience Encyclopedia, Inc., New York, 1951. xvi + 982 pp. 19 × 27 cm. Subscription price, $25.

Elements appear to lead the parade in volume seven, namely: gallium, germanium, gold, hafnium, helium-group, hydrogen, indium, iodine, and the compounds and derivatives of these elements. The inorganic chemist will also appreciate some of the longer sections on synthetic gems, glass, hydrazine, hydrochloric acid, and hydrogen peroxide.

The biochemist will find the longest section (74 pages) devoted to hormones, though there are nine subsections. Other topics of interest to him will be gelatin, glycosides, histamine, and antihistamine agents, as well as those sections of interest to the organic chemist. These will be glycerol, glycols, Grignard reaction, halogenation, heterocyclics, hydrocarbons, hydrolysis, imides, indigoid dyes, indole, and insulin. The analytical chemist will find specific sections on gas analysis, hydrogen-ion concentration, and indicators. The chemical engineer will find particularly useful the sections on heat transfer, gravity concentration, humidity, and instrumentation.

General industrial topics treated in greater detail are furnaces, furs, gas fuels, gas warfare agents, glue, gums and mucilage, hydraulic systems, hydrogenation, ice cream, industrial hygiene, ink, insecticides, and insulation.

Many subject titles now refer to previous volumes and fewer titles refer to volumes yet to appear. The value of this encyclopedia in a reference library continues to grow and its presence has become a necessity.

KENNETH A. KOBE

University of Texas
Austin, Texas

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ANNUAL INDEX—JOURNAL OF CHEMICAL EDUCATION

VOLUME 28, 1951

The annual index for Volume 28 of the JOURNAL OF CHEMICAL EDUCATION is divided into several parts. Authors are listed in three sections: authors of articles, books reviewed, and letters to the editor. The subject index includes both subjects and titles to articles; the books reviewed are listed in a separate section. The principal subject entries are distinguished from titles by printing the former in italics. Portraits, including medals, are indicated by (P).

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2. Manuscript. The manuscript submitted should not have been published elsewhere. The work of the editorial office is facilitated and better service is assured the author when duplicate copies (the original and a carbon) are submitted. Typing of both text and references should be double-spaced, with 2–3 cm. margins. The title of the article should be followed by the name and business or institutional address of the author. If the paper has been presented at a meeting, a footnote giving name of the society, date, and occasion should immediately follow the author's name. The usual editorial customs, as exemplified in the most recent issue of the JOURNAL should be followed as closely as possible.

Galley proof of each article will be submitted to the author. This proof should be carefully corrected and returned promptly to the *Editor's Office*. Page proof will not be submitted.

3. References and Footnotes. In historical and biographical articles and in papers containing only two or three literature references it is desirable that the references be handled as footnotes. Such references should be designated by superior numbers and the reference itself should be placed in the body of the text immediately following the citation and set off by rules.

In articles of the scientific review type each reference to the literature should be designated by a number, in italics (indicated in a typescript by a single underline) and enclosed in parentheses, corresponding in size with the body of the text and set in the line of reading matter. All the references are to be assembled, arranged numerically, and placed at the end of the article under the heading, Literature Cited. The numbers in parentheses are to be placed flush with the margin and if the reference exceeds one line, the succeeding line or lines should be indented. The lists of references should be typed, double-spaced.

When specific citations of previous literature are not made but when it seems desirable to append a general bibliography, it is preferable that arrangement be made alphabetically on the basis of authors' or senior authors' names.

A reference to a periodical should include, in the order named: (1) author's name, (2) name of periodical (use of standard abbreviations designated in the "List of Periodicals Abstracted by *Chemical Abstracts*"), (3) volume number, (4) page (give exact page number at which the point cited is to be found or, if the entire article is the reference, give the page numbers inclusive), and (5) date of publication.

Example:

(1) KRAUS, C. A., AND C. L. BROWN, *J. Am. Chem. Soc.*, **52**, 3690–6 (1930).

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(1) COHEN, J. B., "Organic Chemistry for Advanced Students," 5th ed., Longmans, Green and Company, New York, 1928.

References to explanatory notes such as (1) occasion on which paper was delivered, (2) acknowledgments, (3) definitions of obscure or unfamiliar terms, (4) changes in address, etc., are to be designated in the text by superior numbers in sequence with other footnotes in the article. Such a footnote should be inserted as a separate line (or lines) immediately following the word to which it refers and should be separated from the text by lines.

4. Illustrations. Not all articles lend themselves to illustration, but articles which do should be accompanied by as many pertinent illustrations as possible. The best possible selection of illustrations available will be made by the editorial staff. As a rule, apparatus, particularly if complicated, is best presented by means of line or working drawings. Photographs are sometimes sufficient, however. When both can be obtained, it is generally desirable to include both with the manuscript. Unless lettering can be done by one of the mechanical devices it should be in pencil only.

Photographs should have a gloss finish and should be at least post-card size—larger, if possible. Only prints which are unblurred and which show sharp contrast between light and dark areas can be satisfactorily reproduced. Authors should exercise scrupulous care in crediting photographs which require credit.

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Authors are invited to examine the series of articles by E. M. Hoshall entitled "Chemical Drawing" ["I. Fundamentals of chemical drawing," J. CHEM. EDUC., **11**, 21–3 (1934); "II. Conventional representation of materials and equipment," *ibid.*, **11**, 23–7 (1934); "III. Arrangement of drawings," *ibid.*, **11**, 154–8 (1934); "IV. Charts, graphs, and diagrams," *ibid.*, **11**, 235–41 (1934); "V. Photographs," *ibid.*, **11**, 546–50 (1934).]

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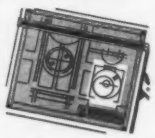

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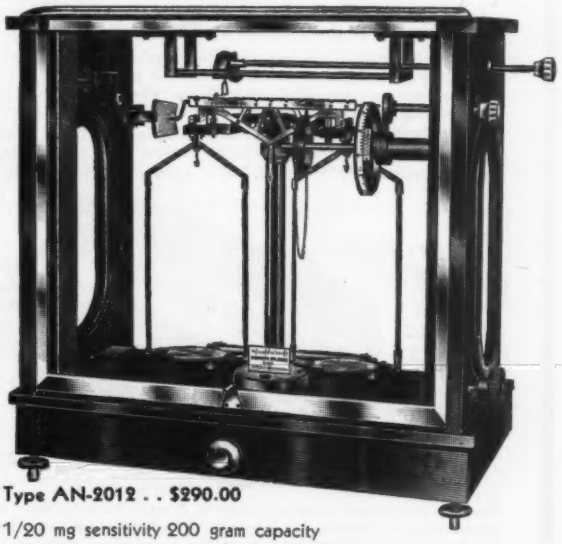

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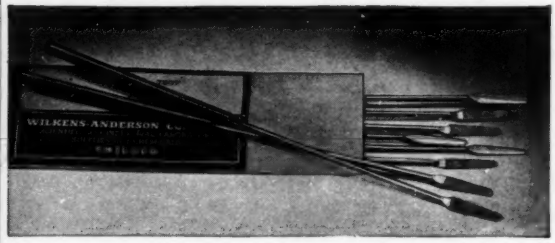

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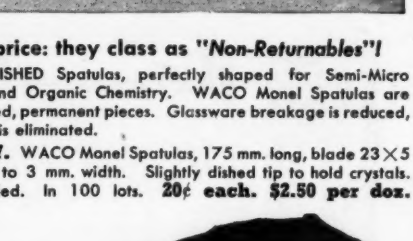

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Squibb type separatory funnels with delivery stems of larger inside diameter are being produced by Kimble Glass, Division of Owens-Illinois Glass Company.

The larger diameter of the stem permits the column of liquid in the stem to break and drain out after the stopcock is shut off, thus facilitating recovery of the liquid in the stem. It is not necessary to empty the funnel and then open the stopcock to obtain this volume. Quantitative separations are more easily and completely obtained.

In addition to this feature, Kimble Squibb type separatory funnels are made with larger neck openings and with more gradually sloping shoulders. Since all sizes hold a considerable amount of liquid over the nominal capacity, safer manipulations are permitted. Both these features permit easier cleaning. Stopcocks and stoppers are of standard taper.

Electronic Parts Catalog

Allied Radio Corporation of Chicago announces the release of a new 1952 catalog containing comprehensive listings of radio, television, and other electronic parts and equipment for classroom, laboratory, and shop. A 212-page publication, it is said to be the largest catalog of its kind. Special emphasis has been placed on the selection of books, materials, training kits, test instruments, parts and equipment required by schools for their radio and electronics training activities.

The new Allied catalog has an expanded section on portable Geiger counters for X-ray and radioactive ore detection in health-hazard applications in laboratories, hospitals, etc.

An extensive rotogravure section is devoted to electronic sound equipment for a wide variety of school applications; especially prepared data for the proper selection of amplifiers, public address equipment, and intercommunication systems for use in classrooms, lecture halls, auditoriums, and outdoor stadiums are included. The "8 to 80 watt" Knight line of public address amplifiers is guaranteed for one year.

The complete section on recording equipment lists the latest disc and tape recorders made by the country's leading manufacturers in the field. All necessary recording accessories also are listed. In addition, the catalog offers a complete selection of FM-AM receivers and tuners, new television units for fringe area reception, communications receivers, and phonographs and accessories for all present types of records.

The 1952 catalog will be sent without charge upon request to Allied Radio Corporation, 833 W. Jackson Blvd., Chicago 7, Ill.

New Organic Chemicals

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Two other new Eastman Organic Chemicals were announced at the same time as additions to Eastman's list of over 3400 organic compounds for research. These are iso-caproamide ($((CH_3)_2CH(CH_2)_2CONH_2$), consisting of nearly odorless white flaky crystals melting at 118–120°C., and 2-amino-6-methylbenzothiazole ($CH_3C_6H_3SC(NH_2):N$), consisting of fine white crystals melting at 136–138°C. The latter is believed to have uses as a dye and drug intermediate, and both have general uses in organic synthesis.

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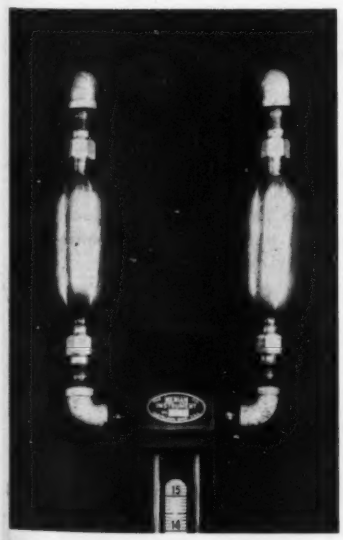

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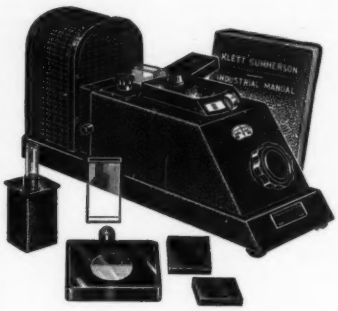

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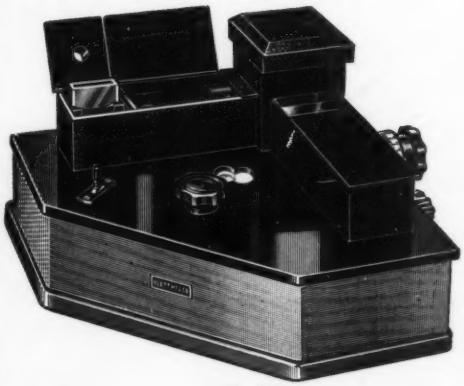

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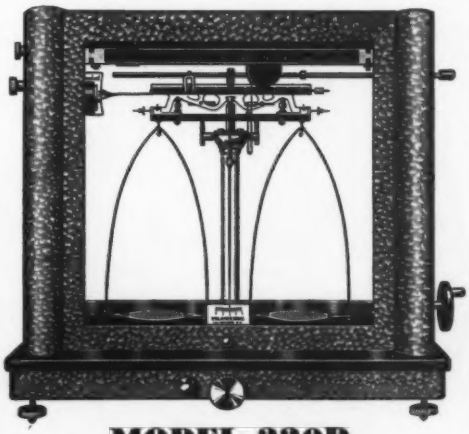

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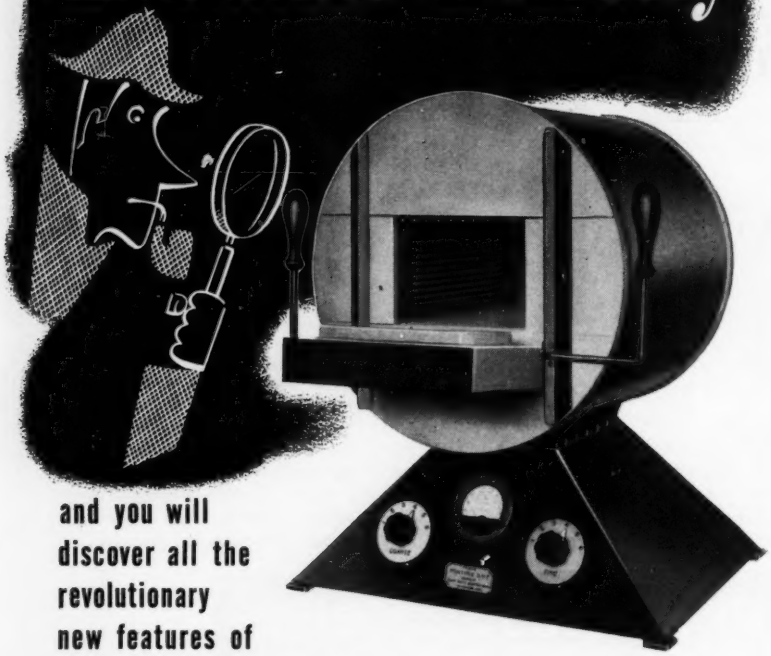

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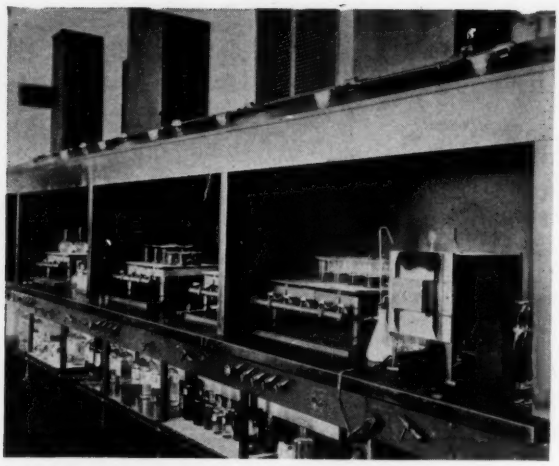

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(1) Ind. Eng. Chem. 25-653 (June, 1933)
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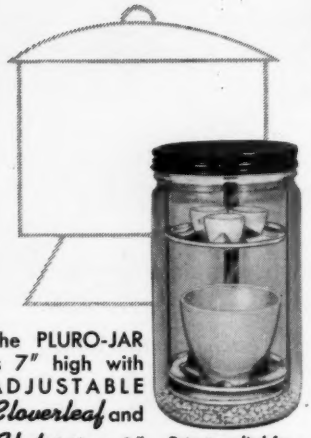

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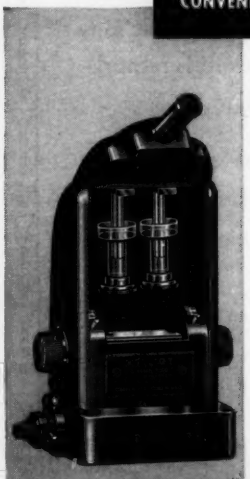

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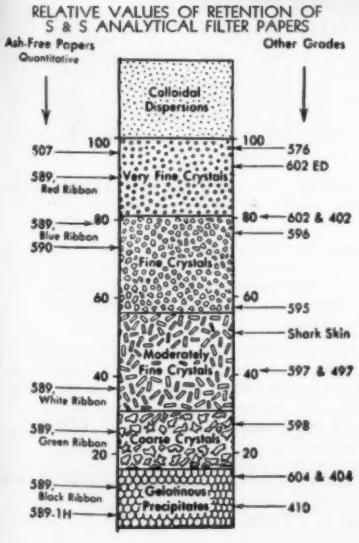

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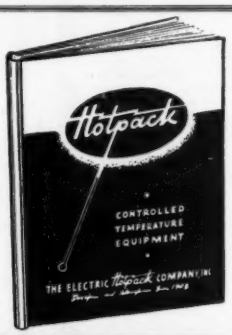

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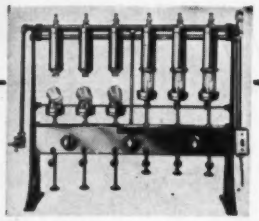

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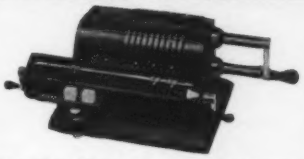

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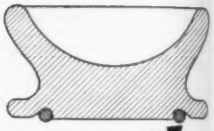

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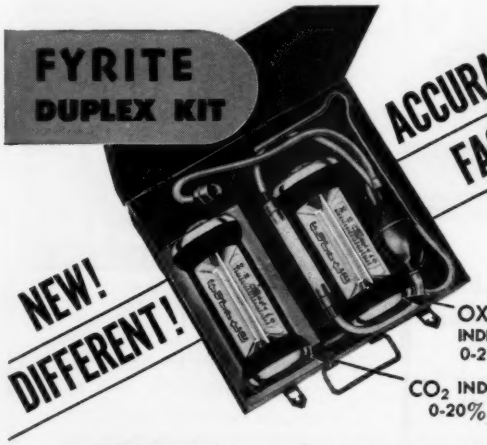

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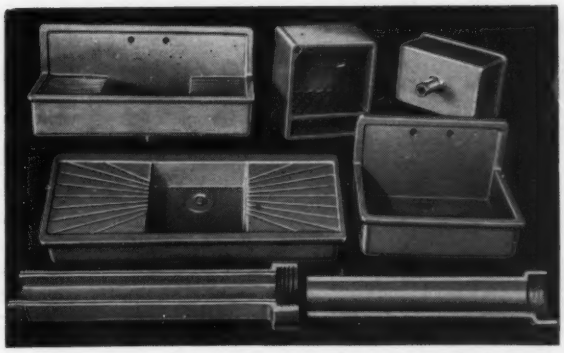

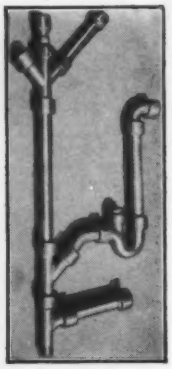

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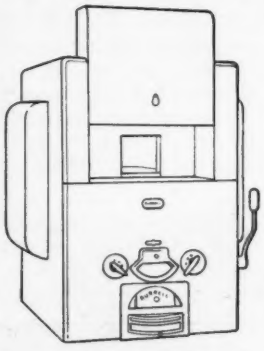

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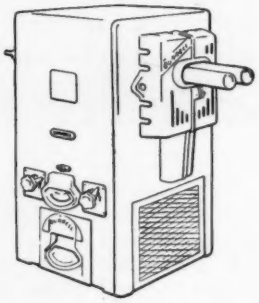

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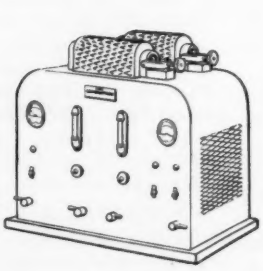

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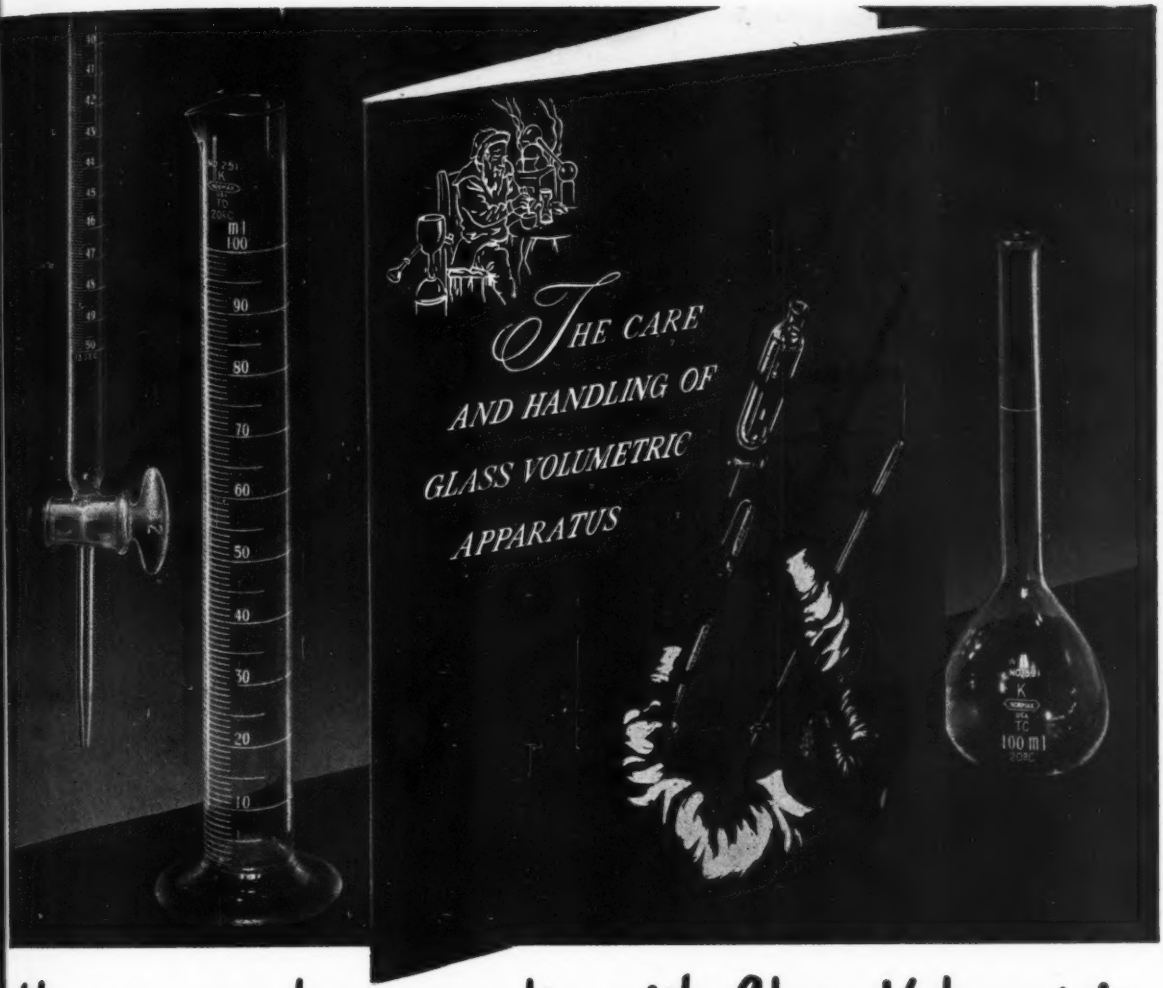

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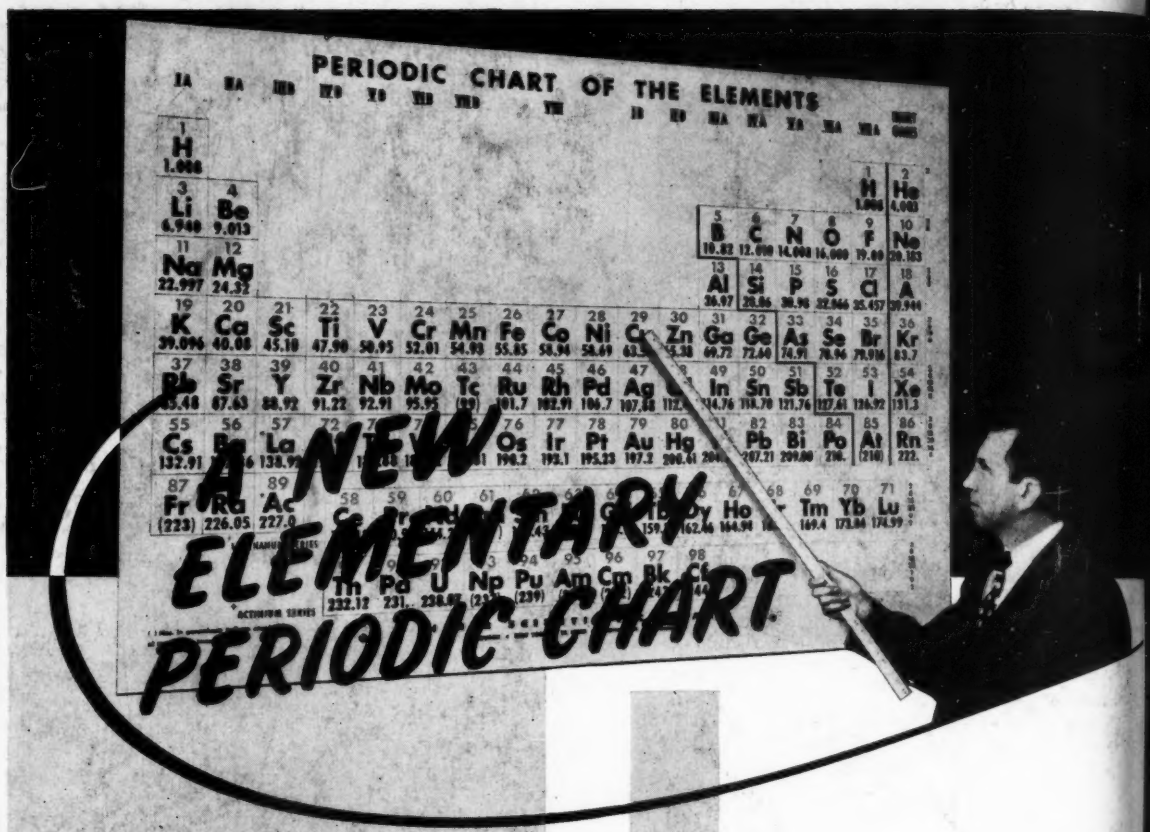

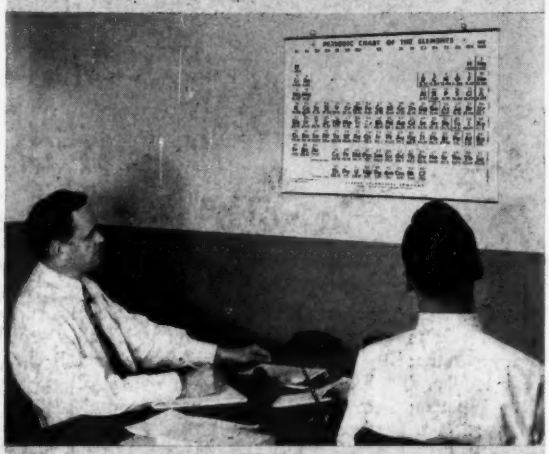

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